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ADVANCED CAPACITORS

HUGHES AIRCRAFT COMPANY
EL SEGUNDO, CALIFORNIA

NOVEMBER 1982

INTERIM REPORT FOR PERIOD APRIL 1980 – JULY 1982

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This technical report has been reviewed and is approved for publication.

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DIELECTRIC FILMS
HIGH ENERGY DENSITY CAPACITORS
HIGH POWER CAPACITORS
DIELECTRIC MATERIALS EVALUATION

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

An experimental development program was conducted to develop and test advanced dielectric materials for capacitors for airborne power systems. High rep rate and low rep rate capacitors for use in pulse-forming networks, high voltage filter capacitors, and high frequency AC capacitors for series resonant inverters were considered.

The initial goal was to develop an improved polysulfone film. Initially, low breakdown strength was thought to be related to inclusions of conductive ———

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particles. The effect of filtration of the casting solution was investigated. These experiments showed that mere filtration was not the entire solution to low breakdown. The film samples were found to contain dissolved ionic impurities that move through the dielectric when voltage is applied and cause enhancement of the electric field. These contaminants enter the film via the resin and solvent, and can be partially removed. However, these treatments did not significantly improve the breakdown characteristics.

A new material, Ultem®, was proposed for use in high energy density capacitors. This new polyetherimide resin has properties similar to polysulfone and polyimide, with improvement in breakdown characteristics and temperature capability.

The technique of casting films on a roughened drum was demonstrated, and found useful in preparing textured films. This is the first step toward a replacement for kraft paper.

For evaluating these materials, a test plan was devised to perform several specific functions. First, improvement can be assessed during the development phase. Second, parameters of particular interest can be identified and evaluated for the capacitor designer. Finally, specific acceptance test criteria can be provided for incoming inspection.

FOREWORD

This interim report presents the progress made by Hughes in developing and testing dielectric materials for capacitors under Contract F33615-79-C-2081, supported by the Aero Propulsion Laboratory at Wright-Patterson Air Force Base, Ohio. Mr. Michael P. Dougherty monitored the program from its inception until recently, when it was transferred to Capt. Gerald Clark. Dr. Robert D. Parker was the Program Manager at Hughes from contract go-ahead until February 1982. Currently the Program Manager is Mr. Robert S. Buritz.

This part of the program (Phase I) was conducted by the Hughes Aircraft Company at its Culver City, California facility. A significant portion of the effort of Task III was contracted to the Schweitzer Division of the Kimberly-Clark Corporation at Lee, Massachusetts. Schweitzer developed and prepared improved film insulating materials, assisted in the definition of the most productive technical approaches, and performed preliminary testing. Dr. W. Selke, Director of Research, Mr. E. P. Bullwinkel and Dr. A. R. Taylor were responsible for the material development and testing conducted at Schweitzer.

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SUMMARY

This interim report contains a description of an experimental development program conducted by Hughes Aircraft Company to develop and test advanced dielectric materials for capacitors for airborne power systems. Four classes of capacitors were considered: high rep rate and low rep rate capacitors for use in pulse-forming networks, high voltage filter capacitors, and high frequency AC capacitors for series resonant inverters. To meet these requirements, existing dielectric materials were modified, and new materials were developed.

The initial goal of the program was to develop an improved polysulfone film with fewer imperfections that could operate at significantly higher electrical stresses. At the beginning of the program, low breakdown strength was thought to be related to inclusions of conductive particles, thin spots, or holes in the dielectric. Many experiments were performed to evaluate the effect of filtration of the casting solution. As these experiments proceeded, it became apparent that mere filtration and attention to the details of maintaining a particle-free film were not the entire solution to the breakdown problem. From a series of experiments, the film samples were found to contain mobile charge carriers, as dissolved ionic impurities, that move through the dielectric when voltage is applied and cause distortion and enhancement of the electric field. It was shown that these contaminants enter the film via the resin and solvent, and that they can be partially removed. As far as developed, however, these treatments did not significantly improve the breakdown characteristics. It still must be determined to what extent the impurities must be removed.

A new material, Ultem®, was proposed for use in high energy density capacitors. This new polyetherimide resin has properties similar to polysulfone and polyimide, with improvement in breakdown characteristics and temperature capability. This material was selected for further study and evaluation.

The technique of casting films on a roughened drum was demonstrated, and found useful in preparing textured films. This is the first step toward a replacement for kraft paper.

Based on the results of the work at Schweitzer, the capacitor designs were revised to include the polyetherimide replacing polysulfone.

For evaluating these materials, a test plan was devised to perform several different specific functions. With this plan, improvement could be assessed during the development phase. Also parameters of particular interest will be identified and evaluated for the capacitor designer during and at the end of the material development phase. Finally, specific acceptance test criteria will be provided for the incoming inspection.

1.0 INTRODUCTION

This document presents the technical approach taken by the Hughes Aircraft Company for the development and test of advanced dielectric materials for capacitors. A significant portion of the program was contracted to the Schweitzer Division of the Kimberly-Clark Corporation, a leading producer of high quality polymer insulating films and electrical grade kraft paper. The dielectric materials used in the previous capacitor development program, conducted by Hughes for AFAPL, were made by Schweitzer. Schweitzer developed and prepared improved film insulating materials, assisted in the definition of the most productive technical approaches, and performed preliminary testing. The several results of this phase of the program are a better understanding of the effect of ionic impurities on breakdown in dielectric materials; a technique for preparing textured films; the selection of a new poly-etherimide resin, Ultem,® for evaluation; and a comprehensive test plan for evaluating dielectric materials.

TECHNICAL INTRODUCTION

A large amount of work has been performed on the development of high energy density capacitors for various airborne applications. Most of the effort has been by capacitor manufac-turers, and there is a scarcity of reported successful results. An energy density* of 210 J/kg (95 J/lb) at 2.5 kV was achieved by Rice, using a patented metallized electrode configuration on a dielectric composed of polyethylene terephtalate coated with cellulose acetate.[1] A pulse-service energy density of 310 J/kg (141 J/lb) at 50 kV was produced by Hoffman and Ferrante, using an unspecified paper/plastic/mineral oil dielectric tested at undisclosed discharge width and repetition rates.[2] In an effort to develop airborne components, Hoffman was able to reach 480 J/kg (218 J/lb) at a few pulses per second, and 264 J/kg (120 J/lb) at moderate repetition rates.[3] These capacitors, which employed a variety of common capacitor materials, were tested in an undisclosed electrical environment. In a similar program, capacitors that demonstrated 10^5 pulse life were made at 110 J/kg (50 J/lb) and 167 J/kg (76 J/lb). The lower density components were tested at repetition rates up to 250 pps in 60-second bursts, while the higher density components were run at rates up to 125 pps in 30-second bursts.[4] Creedon and Fitch reported development of a six-section pulse forming network employing 0.85 μF 40 kV capacitors at 92 J/kg (42 J/lb).[5] The PFN had a nominal 10 μs output pulse and was operated in 30 to 60-second bursts at 125 pps.

In a previous program conducted by Hughes for the Aero Propulsion Laboratory, pulse discharge capacitors were developed for two different services: high rate PFNs (20 μs pulse, 300 pps) and low rate PFNs (20 μs pulse, 50 pps). The results[6,7] showed section energy densities up to 612 J/kg (278 J/lb) for the high rate unit, and a packaged energy density of

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*"Energy Density is commonly ...d in the pulse capacitor field to mean energy storage per unit weight, as J/g or J/lb. The correct quantiti... .. ...ecif ...eight, in kg/J, and energy density, in J/m. Common usage is followed in this section.

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169 J/kg (77 J/lb) for a 2.2 μF 15 kV high rate unit. Section energy densities up to 770 J/kg were observed for the low rate unit.

The intrinsic dielectric strengths of the films available for use in capacitors range from 3.1 MV/cm (8 kV/mil) to 5.9 MV/cm (15 kV/mil), so energy densities in the range 1100 J/kg (500 J/lb) to 3850 J/kg (1750 J/lb) should be possible. Since these numbers are far in excess of those reported in the literature, it is pertinent to ask the reason for the large difference.

Two problems faced in achieving higher energy densities are electrical breakdown and thermal failure. Some capacitors used for pulsed-energy storage fail because corona arising in manufacturing anomalies or material defects eventually punctures the insulation, resulting in a shorted unit. The second failure results from dissipation of relatively large amounts of power in a poorly cooled volume. This failure can take the form of thermal runaway, insulation failure because of very great local hot-spot temperatures, or excessive thermal expansion. This last failure mode is sometimes quite dramatic; the capacitor case suddenly ruptures from the internal pressure.

The AFAPL-funded program previously conducted by Hughes resulted in the elimination of manufacturing defects and many material problems.[8] Failures in high rate units are now observed to occur at fields in excess of 1.97 mV/cm (5 kV/mil) and to be about equally distributed between wearout because of corona at the foil edge and random dielectric failure. Random dielectric failure is caused primarily by dielectric material flaws, such as pinholes, conducting particle inclusions, variations in thickness, and thermally activated flaws.

The objective of this program is to reduce the random dielectric failures by developing materials of higher quality and better dielectric properties than those currently available.

PROGRAM SUMMARY

The program is divided into two sequential phases composed of a total of five tasks. This interim report is a description of the developmental effort and testing conducted by Hughes to accomplish Phase I. A significant portion of the effort of Task III was contracted to the Schweitzer Division of Kimberly-Clark Corporation. A brief summary of the program is given below. The complete statement of work is given in Appendix A.

Phase I—Material Testing and Selection

Task I—Preliminary Material/Dielectric Systems Selection. The most promising existing material candidates for modification and development were determined for the improvement of energy density in each of the four operating regimes. Preliminary candidate dielectric systems were proposed.

Task II—Material Test Plan. A comprehensive test plan was devised for evaluating the materials identified for modification and development. It allows the assessment of improvement in materials being developed, identifies parameters of particular interest to the capacitor designer for evaluation, and provides specific acceptance test criteria.

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Task III—Material Fabricating, Testing, and Final Selection. Test procedures for small and large area breakdown tests were developed. It was shown that breakdowns below the intrinsic level are not caused entirely by inclusions of conductive particles but by dissolved ionic impurities. A new material was proposed for use in high energy density capacitors, and samples were fabricated suitable for use in capacitors. Ample quantities of the new material will be on hand to perform the next phase. Final dielectric systems were recommended. The two required Presentations were made.

Phase II—Capacitor Pad Design and Testing

Task I—Capacitor Pad Design and Test Plan. Capacitor pads will be designed for the four service regimes, and the designs will be documented. The special techniques developed on the previous program will be employed, including constant tension flat mandrel winding and high purity impregnation. Test plans for pad evaluation will be prepared. The test bay developed during the previous program will be used for the pulse capacitor tests.

Task II—Capacitor Pad Fabrication and Testing. In this task, the improvement in energy density resulting from the development of new materials will be demonstrated by fabricating and testing capacitor pads for each of the four service regimes. Failures will be analyzed and documented so that future improvements may be made.

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2.0 TECHNICAL BACKGROUND

Each individual task and accomplishments are presented in this section. First the theory and practice of high energy density capacitor development are summarized.

THEORY

In basic electrostatics, the design of a high energy-density capacitor is deceptively simple. The energy density D is the stored energy divided by the weight:

$$D = CV^2/2W \qquad (2\text{-}1)$$

where C is the capacitance and V the applied voltage. For the most elementary model, that of a two-electrode planar capacitor where edge corrections are neglected, this equation can be written as

$$D = 4.425 \times 10^{-14} \kappa A V^2/(tW) \quad (J/g) \qquad (2\text{-}2)$$

where κ is the relative dielectric constant, A the area of one plate, and t the dielectric thickness. If it is assumed that most of the capacitor weight is in the dielectric, the second equation becomes

$$D = 4.425 \times 10^{-14} \kappa E^2/(d) \quad (J/g) \qquad (2\text{-}3)$$

where E is the electric field and d the density of the dielectric. The assumption made to derive this equation is not particularly bad for large-valued high-voltage capacitors. The resulting simplified equation is helpful in estimating progress during the development process. Clearly the energy density in the dielectric must be greater than that required for the complete component to allow for the weight of the case, termination, and foils.

It is useful to plot Equation 2-3 for different values of κ and d such as might be obtained for various dielectric sandwiches used in high energy density capacitors, as in Figure 2-1. The upper curve represents a polyvinylidene fluoride system that might be used for a DC filter, while the lower curve is representative of a polysulfone system for use where large power transfer is required. Only a moderate field is required to achieve the goal set for the DC filter (220 J/kg), but a larger field is required to achieve the goal set for the low repetition rate pulse capacitors (1100 J/kg).

For detailed design, a more realistic equation that accounts for foil weight is necessary.

$$ED = 4.425 \times 10^{-14} \frac{\kappa E^2}{(d_d + t_f d_f/t_d)} \qquad (2\text{-}4)$$

where d_d and d_f are the density of the dielectric and foil respectively and t_d and t_f are the analogous thickness. This expression takes into account the sometimes appreciable foil weight but ignores margins, tabs, and terminations.

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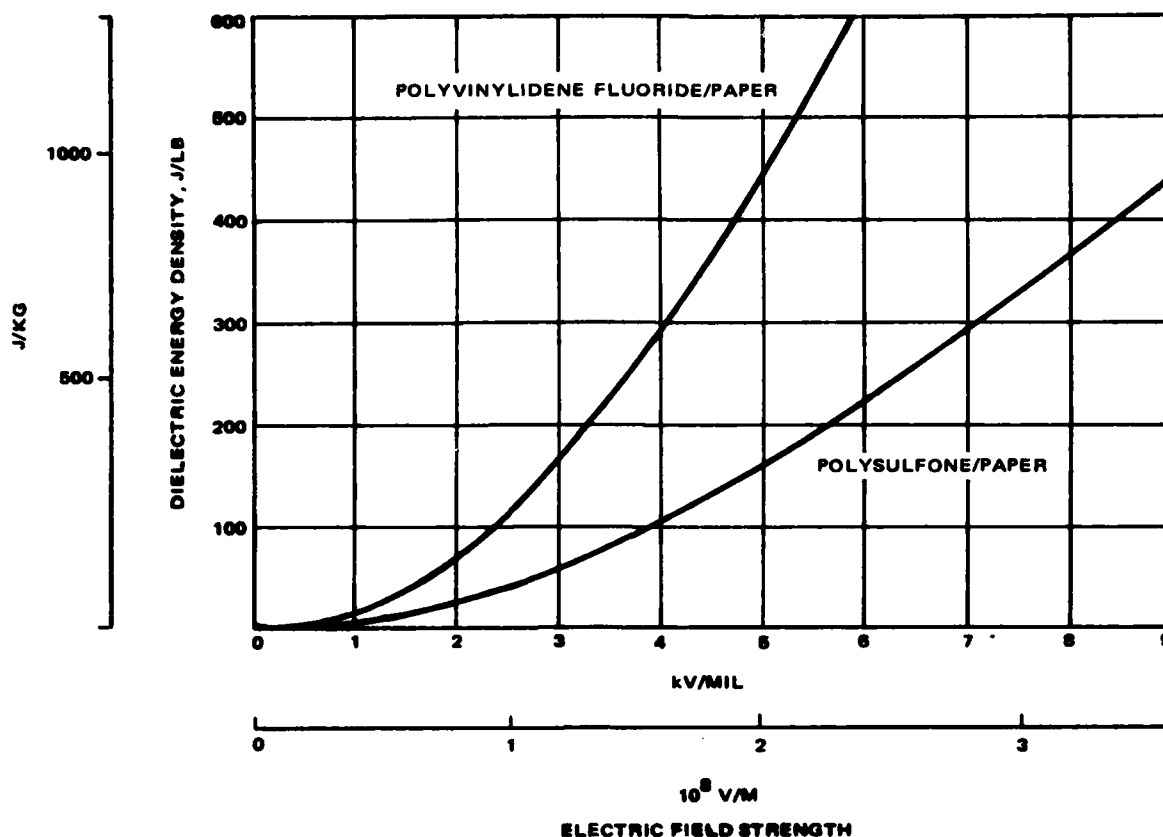
Figure 2-1. Density versus field for two dielectric systems.

It would be simpler if the electric field divided across the various layers in proportion to thickness, but sadly this is not so. While the capacitor sits idle and charged or on DC service, the field division is a DC effect and depends on the volume resistivities of the layers and impregnant. During pulsing or AC operation, the electric field division can be treated as an AC effect dependent on the relative dielectric constants of the individual layers.

As most high power AC and pulse capacitors are made from two different kinds of dielectric, it is sufficient to model a two-layer dielectric. Using Gauss's Law and continuity relations, it is easy to show that the field in dielectric A is

$$E_A = \frac{\kappa_B V}{\kappa_A t_B + \kappa_B t_A} \qquad (2\text{-}5)$$

where A and B are the two different dielectrics, κ the dielectric constant, t the thickness, and V the total applied voltage. The effect of this field division is to drive the electric field in the material with the lower dielectric constant to a value larger than would be expected if a simple average is calculated.

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For the high energy density DC capacitor, it is easy to show that after the component comes to a steady-state condition the fields, in the two layer situation, are

$$E_A = \frac{\sigma_A V}{\sigma_A t_A + \sigma_A t_B} \tag{2-6}$$

where σ is the volume resistivity and the other symbols are given above.

FAILURE MECHANISMS

It is important to understand capacitor failures so that components of higher energy density and improved reliability may be designed. In the following analysis, a general overview of capacitor problems is given.

Electrical Failure

Electrical failure in a large pulse-discharge energy storage capacitor invariably takes the form of corona leading to the establishment of conducting paths, puncture of the insulation, breakdown, and a shorted section. The corona that causes this chain of events is not exactly the same electric discharge one hears and sees on high voltage power lines. Instead, this corona is technically known as "partial internal discharges." An electric field across the dielectric also produces an electric field in the gas that fills any voids, bubbles, or other insulation imperfections. The gas breaks down at a lower electric field than the other dielectric if the characteristic dimension of the imperfection is in the right range. In addition, the field in the gas is somewhat enhanced by the difference between the dielectric constants of the gas and the other insulation. At some electric field a breakdown, or partial internal discharge, will occur in the gas in the void. The breakdown, called corona, is of quite high temperature and tends to burn the insulation.

In a solid dielectric, the repeated breakdowns carbonize the insulation and enlarge the void along the direction of the electric field lines. As time goes on and the void grows, it fires at a lower field strength and high current. An increasingly larger amount of damage is caused, and the situation never gets better. Failure occurs by shorting across the enlarged carbonized void.

The liquid insulations normally used in high voltage capacitors present a different problem, as there is a whole litany of interesting electric-field-related failure modes. In the simplest case, if the dielectric is organic, the corona carbonizes the fluid. The particles of carbon line up along the electric field line and cause repeated breakdowns at decreasing fields and finally failure. For inorganic liquids, the effects depend upon liquid type and sometimes exact composition. They range from decomposition with release of HF gas, to flocculation, to lowered breakdown strength.

Ancillary effects in liquids contribute to electrical failure. Very high electric fields or fields with large divergence, particularly at a point or rough edge of the foil or tab, may cause the spontaneous evolution of gas bubbles either from solution or from decomposition,

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depending on the liquid and temperature. In the presence of the electric field, some fluids react with water to form a dense conductive carbonaceous flocculent. Certain fluids are catalyzed by certain metals at varying reaction rates. Finally, if any particulate contamination is present it will tend to line up along field lines, forming a bridge between the two plates.

Thermal Failure

Failures attributable to thermal effects are found most often when power is transferred at very high rates. The physical phenomenon underlying this form of failure is that all dielectrics dissipate some energy when energy is stored in them, and some when it is released. A measure of this dissipation is a bulk material property called "dissipation factor" or "loss tangent" (power factor is nearly the same thing below a value of 0.01). The dissipation factor DF is defined (ASTM D–150) as

$$DF = \tan \delta = \frac{X_p}{R_p} \qquad (2\text{–}7)$$

where δ is the loss angle, X_p the parallel reactance, and R_p the effective AC parallel resistance. This parameter, different for different materials, varies dramatically with frequency and temperature. As an example, typical curves for three different temperatures for polyvinylidene fluoride (KF film) are shown in Figure 2–2. The dissipation factor measured for a complete capacitor normally is slightly larger than for a dielectric system alone, since I^2R losses in the foils and leads affect the loss tangent. Additionally, since the form of the dissipation factor is as a power loss, the amount of power dissipated depends on the *rate* of energy transfer as well as the total energy stored. This rate effect is important.
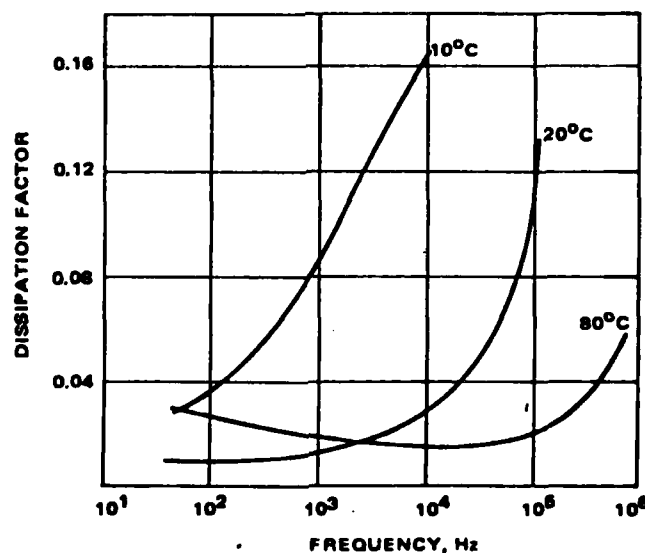
Figure 2–2. Polyvinylidene film dissipation factor versus frequency.

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The failure mechanisms, related either to the temperature rise caused by dissipation of energy in the dielectric or to a thermally-triggered effect, divide roughly into four classes: gross failures caused by thermal runaway, insulation failures caused by the decrease of dielectric strength with increasing temperature, insulation failures resulting from actual decomposition or change of state of the dielectric, and random dielectric failure due to a variety of flaws.

The first group of thermal failures is often the most spectacular: sometimes the oil-filled metal cases actually explode as the increasing temperature causes a large thermal expansion of the oil. A thermal runaway normally requires a material with a dissipation-factor-versus-temperature curve that increases monotonically, as in Figure 2-3. Most plastic films and papers have curves with that characteristic. The usual sequence of events leading to failure is that the component is run at a high power level and gets warm. As it warms up, it dissipates more power, which makes it hotter and which makes it dissipate more power until failure occurs. If the case of the component is not equipped to handle the increasing volume of the hot impregnant, it may suddenly rupture. If not, the high dielectric temperature will cause the dielectric to fail electrically by one of the mechanisms discussed below.
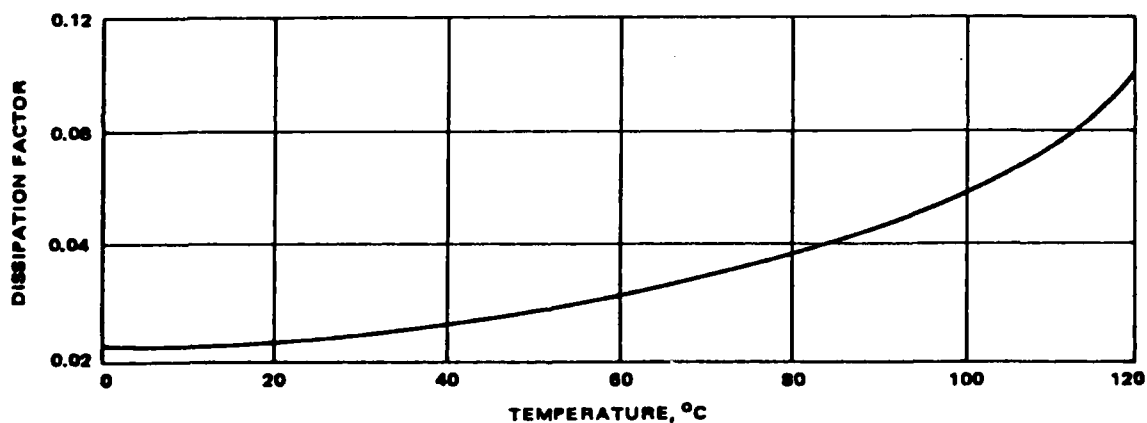
Figure 2-3. Typical dissipation factor versus temperature.

Generally the dielectric strength of a material measured using a standard test (ASTM 149-64) decreases monotonically with temperature. Measurements for several films are shown in Figure 2-4. The problem that this characteristic behavior raises is one of high temperature electric field stability. While the standard test in no way duplicates actual capacitor operation,its results tend to indicate that, at higher temperatures, lower electric fields must be used in given material to obtain the same operating safety factor. Experience with standard film capacitors shows an increase in leakage current and a lower average life at higher temperatures. Usually the ultimate failures are caused by excessive corona and breakdown.

Each material used in a dielectric system has, as an intrinsic property, a temperature above which irreversible physical or chemical changes take place. These changes are different for
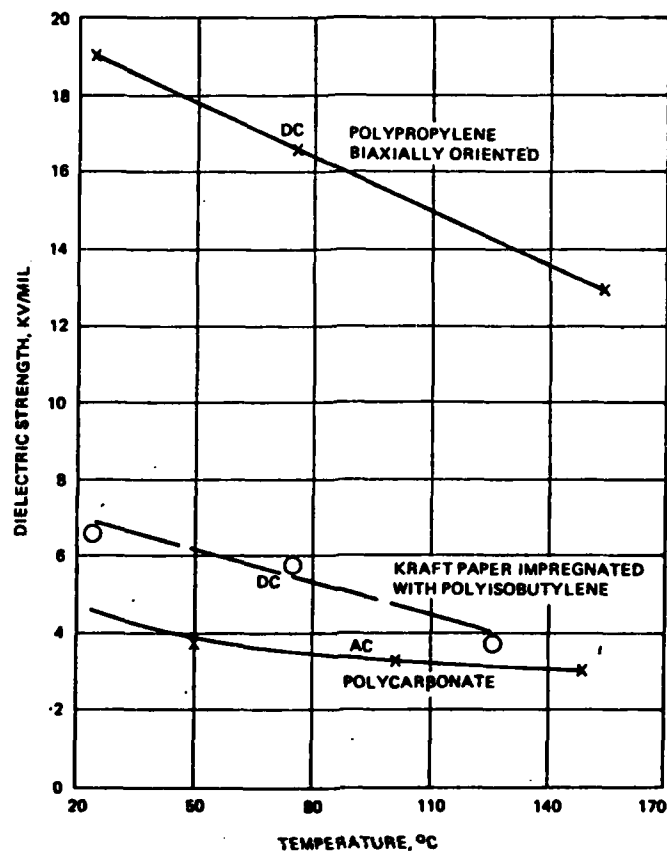
Figure 2-4. Dielectric strength versus temperature
of various capacitor dielectrics.

each material and depend on the particular environment. Polyvinylidene flouride (KF film) in the extruded state shrinks at about 160°C but is chemically stable at 300°C. Cellulose (kraft paper) gives up bound water at about 150°C. This water may then cause hydration of the impregnant or other effects. In high electric fields, some impregnants produce hydrogen gas at high temperatures. Each material is subject to oxidation above a certain temperature. Obviously, the operation of a dielectric system above the service temperature of one of its components will lead to failure, the time of failure depending on the mechanism and the temperature. Both the ambient temperatures and the hot-spot temperatures within the capacitor, caused by power dissipation in operation, are of concern here. A component could be thermally stable (no runaway) but could fail electrically because of this combination of ambient temperature and service temperature rise.

Finally, plastic films fail because of myriad effects grouped under the name "random dielectric failure." Nonuniformities in crystallinity, gel particles, conducting particles, pin-holes, thermally activated flaws, ionic species, and local thickness variations are the causes of random dielectric failures. Some types of flaws, involving ion migration only, cause failures in DC components. Others, such as thermally activated flaws, cause high power AC and pulse components to fail.

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Mechanical Failure

The rhythmic charge-discharge of the capacitor sections can produce mechanical motion at the pulse frequency, particularly if the sections are wound by conventional techniques, as the standard technique produces rather loosely packed sections. When the voltage is applied, electrostrictive forces compress the insulation, which snaps back to its unstressed position on discharge. In severe instances, the capacitors emit clicking noises at each discharge. The mechanical motion causes tabs and connections to fracture and sometimes causes the center of the winding to "walk" out of the section.

PROGRESS ON THE PREVIOUS AFAPL PROGRAM

The initial thrust of the previous program Hughes conducted for AFAPL was that a higher operating field and, therefore, a higher average energy density could be achieved by eliminating manufacturing defects. The most obvious pair of defects—winding wrinkles and the insulation distortion caused by flattening a round pad—were eliminated by the constant tension flat spindle technique. This single improvement allowed the operating field to be increased by 50 percent. Uncontrolled failures resulting from the variability of the impregnation fluid were eliminated by the use of an ultra-pure fluid purification system, which controls simultaneously particulate matter, ionic content, resistivity, water content, and dissolved gas. Since this system allows the consistent preparation of 10^{16} Ω-cm mineral oil and 10^{12} Ω-cm dioctylphthalate, its use has eliminated random oil-caused failures. Also, the dissipation factor of the components has been reduced by as much as 30 percent over those impregnated with nonpurified (10^{12} and 10^{9} Ω-cm, respectively) oil.

These refinements, careful design of the layer system to obtain maximum energy storage in each layer, and other improvements have resulted in a family of 110 to 170 J/kg (50 to 77 J/lb) packaged components that have extremely uniform lifetimes and highly predictable failure mechanisms. When tested to failure, a very repeatable pattern of failure mechanisms emerges. Components that fail in the first half of design life (0 to 50,000 pulses) almost always fail because of random dielectric failure, away from a wrinkle or other mechanical imperfection. Components that survive design life have relatively uniform corona damage along the entire length of the foil edges but usually not enough damage to indicate incipient failure.

A real capacitor can be seen as an ideal capacitor in combination with a series of field and/or temperature activated failure mechanisms. In this program, the problems that occur at lowest field were studied first; then failure mechanisms were eliminated progressively. As each failure problem was solved, the component could be pushed harder until a new failure mechanism, activated by the higher stresses, emerged; at this time, the cycle was repeated. It appeared that random dielectric failure and the general area of dielectric materials were the next problems to be addressed; once a solution was found, the capacitors could be used in a regime in which corona at foil edges was the principal problem.

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3.0 TASK I—PRELIMINARY MATERIAL/DIELECTRIC SYSTEMS SELECTION

In this first phase of the program, the dielectric materials requirements were determined, candidate materials and improvements were selected, and improved materials were fabricated and tested. This phase was divided into the three tasks discussed in this section. Schweitzer Division of Kimberly-Clark assisted in the performance of Tasks I and II and performed substantially all of Task III.

The program conducted by Hughes before the present program resulted in the development of lightweight high-repetition-rate pulse discharge capacitors employing polysulfone, kraft paper and dioctylphthalate as a dielectric system. These capacitors, designed to a performance specification similar to that of paragraph 4.3.1 of the Statement of Work of the present program, failed by two different mechanisms:

1. Random dielectric failures (RDF)

2. Corona at foil edges.

The first failure causes both infant mortality and wearout, while the second is primarily a wearout mechanism. The problem of corona at foil edges has been discussed in the literature and was addressed during the previous program, although only small foil specimens were actually fabricated and tested. The improvements sough in this task were, therefore, in the reduction of RDF in the dielectric system.

CANDIDATES FOR IMPROVEMENT

The quality and energy density of a capacitor dielectric system might be improved by several methods. An extensive discussion of a wide variety of approaches was included in the proposal, per the requirements of paragraph 4.1.1.1 of the Statement of Work. Only those techniques seriously pursued in the first phase are discussed in this section.

Higher Quality Films

The practical operating stress of film dielectrics, in general, is far below the "intrinsic dielectric strength" of the polymer. But experiments demonstrate that, at least in tests on small areas where the likelihood of encountering an electrical weak spot is slight, breakdown stresses in excess of 600 volts per micron can be attained. In spite of this fact, not much effort has been expended in the production of film with fewer weak spots because historically such films have been used in metallized capacitors in which the weak spots can be eliminated easily by "clearing." Experiments have shown that these electrically weak spots may be the result of conducting particles, voids, pinholes or low basis weight (low mass/area).

The initial goal of this research program was to develop polysulfone film that, by reducing the frequency of electrical imperfections, can operate at electrical stresses significantly above those currently employed. Polysulfone film was chosen for study because it has

good dielectric properties over the temperature range of interest, is compatible with a range of dielectric fluids used for impregnating capacitors and, most important, is already of a quality that the goals envisioned for the program were reasonable.

The first step in this program was to identify, by non-destructive testing, the most frequently encountered causes for low electrical strength. These techniques have been used in the past by the Schweitzer Division for identifying causes of breakdown in both films and in paper. Then, improved experimental films were to be made on the casting equipment. Improved techniques for the filtration of casting solution (dope) were explored during this phase in an attempt to reduce the number of conducting particles *in* the film. Also, filtration of drying air was explored to reduce the number of particles deposited *on* the film. Large area specimens of these films were tested for electrical breakdown to assess the progress. The final step was scaling up the laboratory developments to production size equipment. The influence of hot stretching on impairing dielectric strength was considered carefully at this point.

Denser Kraft Paper

A separate subject in the development of improved dielectrics for high energy density capacitors is the use of a denser, thinner kraft capacitor tissue than currently used. Several advantages can be realized by such a approach. First the entire dielectric pad becomes thinner, and higher capacitance is obtained at equivalent basis weight. Second, a denser paper will produce very thin layers of impregnating liquid. The dielectric strength of liquids, while generally lower than that of dielectric solids, can be very high if the layers are thin enough. Finally, the paper substance, because of its higher dielectric constant, transfers much of the electrical stress to the dielectrically stronger layers of film. Hence, a more dense paper will mean even more stress will be transferred to the film.

One problem encountered in the previous AFAPL program was the optimization of the design of AC and pulse discharge capacitors to obtain the maximum safe energy storage in each dielectrical layer (film, paper, oil). A typical field distribution for a very high energy density component is given in Table 3-1. The field distribution in an AC or pulse component depends on both dielectric constants and thicknesses of layers, so that the use of a denser, thinner kraft will shift more stress to the polysulfone.

TABLE 3-1. FIELD DISTRIBUTION IN PULSE
DISCHARGE COMPONENT

Material	Field, V/mil	Breakdown Field, V/mil	Ratio*
Kraft	3430	4500	0.76
Polysulfone	5266	7500	0.70
Oil	2761	400	6.9
*Operating field/breakdown field			

The dielectric constant of kraft is somewhat of a volumetric average number. The figure for cellulose is 6.3, while that for dry kraft is in the range of 3.5 to 5. This is because the kraft is not smooth; therefore, some of the volume between measuring electrodes fills with air (or, in a capacitor, the oil used for impregnation). A denser paper has more kraft per unit volume and fewer spaces. Then there will be less fluid, and the dielectric constant of the paper-fluid assembly will increase (if the dielectric constant of the fluid is below 6.3). Also, because there are fewer spaces, the chance for electrically weak spots is reduced; therefore, the average dielectric strength is improved. All of these factors aid in achieving a more favorable field balance—one in which each material is stressed at the same percentage of its breakdown stress.

Then several advantages accrue:

1. Thinner fluid layer (higher dielectric strength)
2. Higher dielectric constant
3. Higher dielectric strength (paper)
4. Better field balance (AC and pulse).

Kraft Paper Replacement

As venerable an insulation as it is, kraft paper has three serious drawbacks:

1. Shrinkage (non-isotropic)
2. Moisture absorption
3. Low decomposition temperature.

Therefore, it might be interesting to investigate a replacement that performs the same functions as kraft but that does not have as many bad features.

Strictly speaking, the common explanation that kraft paper acts as a wick or a medium enhancing impregnation is fallacious, since the paper is actually nonporous and nonwicking to any liquid that is not aqueously based. Kraft first functions as an electrical barrier, as any plastic film would, raising the insulation breakdown voltage from 400 V/mil for fluid to about 4500 V/mil. By comparison, fluid filled porous felt, which acts as a wick but not as a barrier, breaks down at about 700 V/mil. Second, the kraft provides a means for defeating the electrostrictive failures. Any replacement must seek to do both these tasks.

Kraft paper can be replaced in one of two ways: the impression of a matte surface onto a plastic film or the treatment of the film to enhance wettability. The first alternative was explored extensively.

A matte surface can be impressed onto the plastic film easily, at least in principle, since the polysulfone (and most other candidate films except polyvinylidene fluoride) is solution cast. A casting drum would have to be provided with a pattern engraved on the surface, and the pattern would be replicated faithfully on the surface of the film.

Film Lamination

Capacitors are normally made with a multiplicity of layers, to greatly reduce the possiblity of failure due to a through hole or conducting flaw in a single film. It is clearly also possible to make a laminated film, in the manner of a flat cable, which would greatly reduce the through holes but that would have the advantage of winding as a single film. This material would have a substantially higher dielectric strength than a single film of the same thickness. It was thought that it might be possible to make a two-layer lamination in about the same thickness presently used (7 to 13 μm).

High Dielectric Constant Impregnants

A number of fluids with dielectric constants exist in the range 6 to 12. Two are well-characterized, and the remainder are either little used or new experimental materials. Fluids with large dielectric constants exist; the most common example is water. In general, however, these fluids are useful only in certain specialized situations and are not applicable for military capacitor service. The properties of some of the possible fluids are summarized in Table 3-2.

TABLE 3-2. PROPERTIES OF IMPREGNANTS

Dielectric Liquid	Density, gms/cm^3	Freezing Point, °C	Boiling Point, °C	Dielectric Constant (1 kHz)	Dissipation Factor (1 kHz)	Volume Resistivity, ohm/cm	Electrical Breakdown Strength, volts/mil	Thermal Conductivity, cgs units
PCB Arochlor 1242	1.39	−19	325–366	5.8	0.001	1×10^{10}	550	28×10^{-5}
Alkylated Benzene DO-100	0.57	−50		2.1	0.002	1×10^{15}		25×10^{-5}
Mineral Oil GB-100	0.51	−45		2.2	0.0005	1×10^{14}	390	30×10^{-5}
SF-1050	0.92	<−100	261	2.48	0.005	1×10^{15}	480	27×10^{-5} at 20°
Dioctylphthalate	0.99	<−45		5.7	0.001	1×10^{11}		24 at 150°
Castor Oil	0.95	<−15		4.7	0.001			
MCS 1238	0.91	−45		5.9	0.001	1×10^{15}	400	30×10^{-5}
High Wetting X2-2528	0.92	−70	248	2.7	0.0001	1×10^{15}	700	28×10^{-5}

The polychlorinated biphenyls (PCBs) have been widely used as capacitor fluids. Formerly available as Arochlor, Pyranol, or Inerteen, they were not considered for this program because their production and use have been banned in the United States after 1978.

Dioctylphthalate was selected as a candidate fluid for its potentially high energy density storage properties. This material was one of four selected for capacitor use in the previous AFAPL program, and extensive contamination and compatibility experiments have been conducted. This organic ester is being used commercially in transformers and capacitors as a replacement for the unavailable PCBs. The ester offers a relatively high dielectric constant

and a low density. The power factor of the organic esters is relatively high at low frequencies and low at high frequencies. The DC resistivity is characteristically low. However, both resistivity and power factor are affected by the chemical purity of the ester. To take maximum advantage of the desirable properties of dioctylphthalate, the fluid must be highly refined to achieve and maintain a very low acid number and a very low dissolved water content. As a generic class, the organic esters are more vulnerable to hydrolysis by water than are the hydrocarbons and silicones.

A variety of modified mineral oils, such as Monsanto's MCS 1238, have dielectric constants above six. These fluids are prepared by the addition of a high dielectric constant modifier to a suitable mineral oil. With this technique the fluid can be tailored, within limits, to achieve field balance within any given structure. MCS 1238 was made by using a sulfone additive and had a dielectric constant of 5.9 to closely match kraft paper. Since the sulfone has a dielectric constant of about 27, however, any dielectric constant in the range 2 to 27 can be made. Above about 10, unfortunately, the other electrical properties of the mixture are not ideal for energy storage capacitors. One problem with mixtures of any type (even mineral oil) is that vacuum degassing processes are made difficult by preferential distillation of one component. Reliable experimental data on the MCS 1238 class fluids are not presently available.

EXPECTED IMPROVEMENTS

Primarily as a result of the expected improvement in the polysulfone film, an expected 21 percent improvement in energy density was originally estimated.

If the number of breakdowns per unit area of a large piece (~ 100 cm^2) of film is measured as a function of applied field, a curve such as that shown in Figure 3-1 is obtained. The
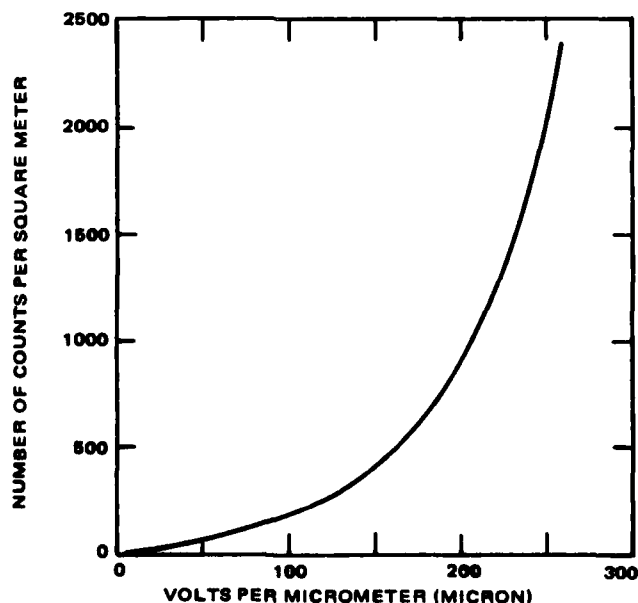
Figure 3-1. Typical large area breakdown test results.

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failures in the lower field part of the curve represent the defects that cause the capacitor structure to fail at a field well below the knee of the curve. Initially it was thought that the steps described above would result in the elimination of many of those "defects," causing the curve to be essentially at the zero count level until the knee was reached. It was thought that an improvement of the operating electric field of at least 10 percent would be achieved. Because the energy density goes as the square of the electric field, a 21 percent improvement was projected.

PRELIMINARY CANDIDATE SYSTEMS AND ESTIMATES

Preliminary pad designs and estimates of average dielectric constant, average stress, and energy density of the pad are given in this subsection. These designs represent minor changes, in most cases, to designs previously developed; therefore, some data, particularly weights, are used from previous designs.

High Repetition Rate Capacitor

This design is similar to that found in AFWAL-TR-80-2037, page 86, labelled design A.

Total layers dielectric	: 5
Layers polysulfone	: 2
Polysulfone thickness	: 6μ (24 gauge)
Layers kraft paper	: 3
Kraft thickness	: 7.6μ (0.3 mil)
Foil	: Aluminum, 6μ
Dielectric fluid	: Dioctylphthalate

For this configuration, the following estimates can be obtained:

Operating voltage per pad	: 7500 V
Average field	: 5068 V/mil
Average dielectric constant	: 4.29
Energy density	: 0.28 J/g (128.6 J/lb)

Low Repetition Rate Capacitor

This design is similar to the above design. The repetition rate is too high to allow the use of polyvinylidene fluoride.

Total layers dielectric	: 5
Layers polysulfone	: 2
Polysulfone thickness	: 6μ (24 gauge)
Layers kraft paper	: 3
Kraft thickness	: 1-7.6μ, 2-10.2μ
Foil	: Aluminum, 6μ
Dielectric fluid	: Dioctylphathalate

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Estimates are:

Operating voltage per pad	:	8000 V
Average field	:	5063 V/mil
Average dielectric constant	:	4.32
Energy density	:	0.28 J/g (128.3 J/lb)

DC Capacitor

The ripple in this component is low enough so that the principal failure mechanism will be electric-field-driven rather than thermal. Then polyvinylidene fluoride can be used.

Total layers dielectric	:	5
Layers PVDF	:	3
PVDF thickness	:	9μ (35 gauge)
Layers kraft paper	:	2
Kraft thickness	:	7.6μ (0.3 mil)
Foil	:	Aluminum, 6μ
Dielectric fluid	:	Dioctylphathalate

Estimates are:

Operating voltage per pad	:	8333 V
Average field	:	4273 V/mil
Average dielectric constant	:	7.19
Energy density	:	0.3375 J/g (152.8 J/lb)

High Frequency AC Capacitor

This component is difficult because of the combination of military temperature range (although no specific temperature specification is given in the SOW) and very large power throughput. The military temperature range rules out materials such as styrene and polyethylene, which have the lowest dissipation factors, while the high power throughput argues for a very low dissipation factor. Accordingly, kraft paper is not used, and a very low loss impregnant is selected.

Total layers dielectric	:	3
Material/thickness	:	Polysulfone/8μ
Foil	:	Aluminum, 6μ
Dielectric fluid	:	Silicone

Estimates are:

Operating voltage per pad	:	1500 V
Average field	:	2343 V/mil
Average dielectric constant	:	3.33
Energy density	:	0.05 J/g (21.3 J/lb)

4.0 TASK II—MATERIAL TEST PLAN

INTRODUCTION

As described in the statement of work, the Material Test Plan (presented in its entirety in Appendix B) is intended to perform several different specific functions.

1. To assess the improvement in the materials being developed during the development phase
2. To identify and evaluate parameters of particular interest to the capacitor designer, during and at the end of the material development phase
3. To provide specific acceptance test criteria, to be applied as an incoming inspection to the materials as produced in their final forms

Three specific test sequences are envisioned: developmental testing, parametric charaterization, and acceptance testing. These are explained in the remainder of this section.

DEVELOPMENTAL TESTING

Developmental testing is performed during the work aimed at improving the characteristics of insulating materials. Particularly difficult or delicate tests may be included, tests that normally would not be performed during incoming inspection. Usually complete parametric characterization would not be performed at this stage unless new materials or new chemical compounds were being used.

Developmental tests are performed at the discretion of the development engineer to show improvement. Only the properties expected to improve normally would be tested.

PARAMETRIC CHARACTERIZATION

Tests to completely characterize the material may be performed during the development phase but most certainly will be performed at the end of the development phase. The purpose of this battery of tests is to give information to the capacitor designer. Therefore, important properties at the anticipated operating points in actual devices will be measured.

ACCEPTANCE TESTING

Acceptance tests are performed to make reasonably certain that the insulation material is as ordered, to detect gross defects, and to ensure that the material has been properly packaged and not damaged in shipment. For these reasons, neither complete parametric characterizations nor difficult developmental tests will be performed.

TEST PROCEDURES

Procedures for testing are organized into separate groups for films, oils, and papers. Each group is divided further into electrical tests, physical properties, chemical properties, packaging, etc. The test procedures for films are summarized in Table 4-1. Test procedures for oil are

summarized in Table 4-2, and test procedures for papers are summarized in Table 4-3. As noted in the Test Plan, displayed in Appendix B, the procedure for each test is specified in detail or referred to an ASTM specification.

TABLE 4-1. TEST PROCEDURES FOR FILMS

ELECTRICAL TESTS	ROLL FORMATION AND CHARACTERISTICS
Dielectric Constant	
Dissipation Factor	Cores
Surface and Volume Resistivity	Roll Workmanship
Dielectric Strength	Quality
Gross Flaw	Width and Diameter
Fine Flaw	Splices
TESTS FOR CONTAMINANTS	Marking
Surface Contamination	**PACKAGING**
Residual Solvent	Wrapping
Moisture Absorption	Outer Package
PHYSICAL AND MECHANICAL TESTS	General Requirement
Film Thickness	
Film Density	
Tensile Strength at Break	
Elongation at Break	
Shrinkage	

TABLE 4-2. TEST PROCEDURES FOR OILS

PHYSICAL PROPERTIES	CHEMICAL PROPERTIES
Viscosity	Neutralization Number
Flash and Fire Points	Water Content
Pour Point	Inorganic Chlorides and Sulfates
Specific Gravity	Oxidation Stability
Weight per Gallon	Oxidation Inhibitor Constant
Interfacial Tension	Gassing
Color	**PARTICULATE CONTAMINATION**
Visual Examination	
ELECTRICAL PROPERTIES	
Dielectric Constant	
Dissipation Factor	
Dielectric Breakdown Voltage	
Volume Resistivity	

TABLE 4-3. TEST PROCEDURES FOR PAPERS

ELECTRICAL PROPERTIES	CHEMICAL TESTS
Dielectric Constant	Aqueous Extract Conductivity
Dissipation Factor	Soluble Chlorides
Dielectric Strength	Acidity—Alkalinity—pH
Conducting Paths	Moisture Content
Fine Flaws	
	ROLL CHARACTERISTICS AND PACKAGING
PHYSICAL TESTS	Cores
Paper Thickness	Roll Workmanship
Apparent Density	Width
Holes and Felt Hair Inclusions	Marking
Tensile Strength and Yield	Packaging
Properties	
Air Resistance	

REQUIREMENTS

The test requirements are given in tables that give the required test points and acceptance values for each test. A separate table is given for each test sequence. Not all tests are intended to provide a "go-no go" criterion but rather to provide parametric values of the various material properties of interest to the capacitor designer. For these tests, the word "Report" appears in place of a specific set of limits for acceptance and rejection.

USING THE TEST PLAN

The test plan is divided into films, oils, and papers. The test procedures (specifications) are listed according to each group. Most tests are performed per ASTM specifications. Special tests are explained and defined. The test requirements are summarized in the tables according to test sequence; i.e., developmental, parametric characterization, or acceptance tests. To use the test plan, the test sequence must be picked and then the appropriate tests selected from the table of test requirements.

The Material Test Plan is a flexible, comprehensive document. It can be used effectively

1. To assess improvement in materials being developed

2. To provide data of interest to the designer during the development phase.

Although the test plan is not organized along the lines of a military specification, it could be used easily to produce a military specification that would

• Furnish inprocess controls for manufacturing

• Provide acceptance tests for production materials.

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5.0 TASK III—MATERIAL FABRICATION AND TESTING

INTRODUCTION

The manufacture of capacitors with high energy density places severe demands on both the dielectric material and the workmanship in the capacitor itself. This latter problem has been addressed in previous work at Hughes. The purpose of the present work was to develop improved dielectric materials. Since the energy density in a capacitor varies with the square of the applied voltage, the most effective way of building high energy density capacitors is to use dielectric materials that will withstand high voltage stress without breakdown.

Previous work at Hughes on a wide variety of available dielectric materials showed that polysulfone film had the necessary combination of properties to make it suitable for further development. These properties included stable dielectric constant over the range of temperatures and frequencies of interest and a low dissipation factor over the same range of temperature and frequency. Therefore the primary goal of this study was to produce a polysulfone film with a significantly higher dielectric breakdown strength than standard commercial film. Further, the technical effort could be redirected toward other materials that may increase the likelihood of achieving increased dielectric performance.

It is fair to ask why commerically available polysulfone dielectric film does not have a dielectric breakdown strength high enough to be useful in high energy density capacitors. The answer is primarily a practical one. By far the largest use of such films is in metallized film capacitors in which defects in the film are "cleared" when voltage is impressed, leaving a tiny hole in the dielectric and a small area around the hole where the thin metallized layer has been vaporized. This process removes imperfections in the film without harming its dielectric properties. Such a process does not pertain to film/foil constructions as envisioned for high energy density capacitors where any breakdown in the capacitor would result in a short circuit. In addition, typical metallized film constructions are used at significantly lower fields than envisioned for high energy density capacitors. Thus, no commercial reason has been found for providing polysulfone film with high dielectric strength.

The intrinsic dielectric breakdown strength of all dielectrics (polysulfone included) is well above that observed in practical applications; i.e., breakdown stresses of 700 to 800 V/μm can be observed under special conditions and these levels of stress are inherent or "intrinsic" to the dielectric. At the beginning of the present program, it was believed that breakdowns below this level were related to inclusions of conductive particles, thin spots or holes in the dielectric. As shown in this section, while these defects are undoubtedly important in dielectrics, other phenomena are also important, especially at high stress, and remain poorly understood.

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The various test apparatus and procedures developed for small and large area destructive breakdown tests, attempts at developing a nondestructive breakdown test, and equipment for measurements of conductivity on various films are described here. Then, the experimental results for large and small area breakdown tests and the conclusions are given. Finally, the recommended dielectric systems are discussed.

VOLTAGE BREAKDOWN TESTING

At the beginning of this work, a strong belief was that the key to improving the large area dielectric strength properties of polysulfone films would involve lowering the density (number per unit area) of possible defects (Figure 5-1). Such a belief immediately suggests that it would be extremely valuable to have a method of determining the actual density of these individual defects so that the efficacy of various process modifications could be directly known and not merely inferred. Accordingly, much effort was expended on developing nondestructive voltage breakdown testing (VBT) methods that would survey large film areas continuously. This work was in two parts. In the first, electrode systems would be developed that would give essentially line contact across a moving film web without causing mechanical damage to the delicate web. (By the provision of indexing markers, portions of film carrying the line locus of breakdown could be removed from the web and subsequently scanned along the line to exactly locate the breakdown site.) The second part was circuit development that would remove or reduce the enormous power dissipation at the breakdown site. (This dissipation is responsible for the destruction of any defect evidence in ordinary VBT.)
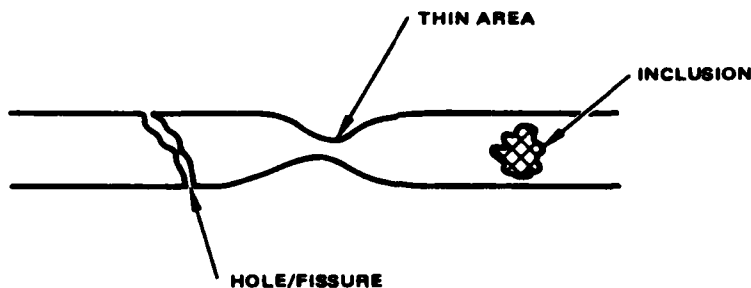
Figure 5-1. Density of defects in polysulfone films.

While success was achieved in developing suitable electrodes, the second part that involved preservation of the breakdown site proved intractable. Before these matters are discussed, one quick, simple method of large area VBT employed over the years is described. This static method merely involves laying a film sample on a large, polished brass plate, that acts as one electrode. Next, a web of metallized film, metal side down, is carefully placed over the sample to serve as the second electrode.

When voltage is applied across these two electrodes, electrostatic forces in conjunction with the limpness of the two films tend to squeegee out bothersome air interfaces and give very intimate electrode-film contact. Whenever a voltage breakdown occurs in the test sample, the metallized film electrode "clears." This clearing, the result of volatilizing of the

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metal adjacent to the breakdown site, not only gives a permanent visual record but also automatically disconnects the site from the remaining test area so that the test can be continued without the need for external intervention.

Apart from the slight bother of laying up samples, this test is extremely useful. Indeed, this method was used for most of the large area testing (LAT) reported. Nevertheless, since the method involves volatilizing metal, it certainly is not the basis for nondestructive VBT of polymer films.

Returning now to the development of line contact electrodes suitable for testing moving film webs, the final design evolution is shown in Figure 5-2.
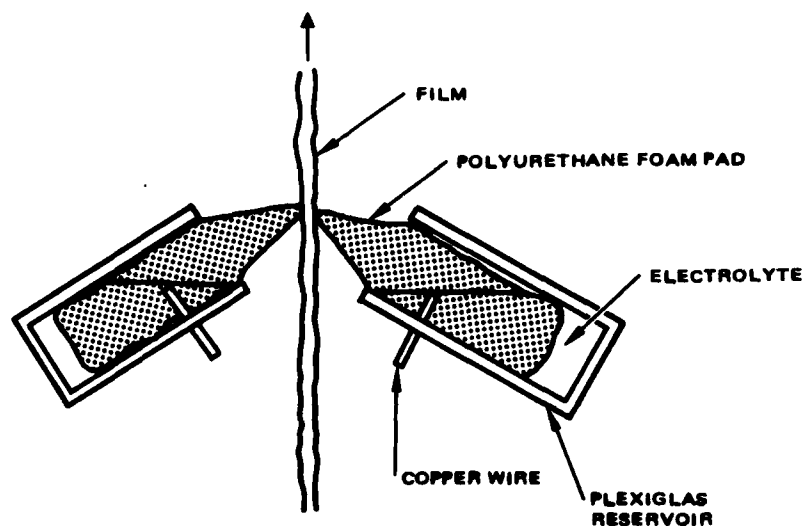
Figure 5-2. Aqueous line contact electrodes.

In this arrangement, the actual electrical contact to the film is achieved by an aqueous electrolyte solution maintained *in situ* by various surface tension and electrostatic forces involving this solution, the foam pad wicks, and film under test. While the polysulfone film surface is sufficiently hydrophobic so that a discontinuous (beaded) electrolyte film is deposited on the web as it exits from the line contact, these electrodes must be used carefully to provide sufficient dead time following a breakdown so that false surface flashovers are avoided. In general, this dead time (i.e., voltage removed) should be long enough so that at least 1 cm of film has passed through the electrode system.

The favorite electrolyte has been $0.1\underline{F}$ Cu (en)$_2$ SO$_4$. In contrast to ordinary CuSO$_4$ solutions, this ethylenediamine complex ion avoids fouling the copper terminal with various copper oxides. Additionally, the strong violet color of this ion is helpful in aligning the electrodes. Experiment has shown that the bulk conductivity of polysulfone film is not enhanced in the presence of this aqueous electrolyte.

The use of the wedge shaped pads (available in most hardware stores as paint applicators) permits line breadth contacts down to ca. 1 mm so that interelectrode capacitances of only 25 pF per cm of electrode width are achievable with 10 μm polysulfone film. (The importance of this capacitance will become clear below.) The electrical contact with the film by these conformable liquid electrodes is equivalent to that obtained with evaporated metal electrodes, i.e., no disturbing air gaps are present.

The principles of nondestructive VBT are simply stated—detect the very first beginnings of voltage collapse across the sample and then, as quickly as possible, disable or crowbar any energy sources that can promote any future growth or duration of the breakdown process with the reasonable hope that the sample will be returned to its normal insulating state undamaged. (These energy sources are invariably various stray capacitances that are fully charged to the particular test voltage at the time of breakdown initiation.)

A reasonable circuit for performing these tasks is shown in Figure 5-3 where a small thyratron crowbar can be triggered into full conduction in approximately 10 to 20 ns by overdriving its grid with a fast discriminator-trigger circuit (avalanche transistors). The discriminator is provided to distinguish a true breakdown event from minor surface discharges that occur when using the aqueous line electrodes described above. The input to this discriminator is generated by current pulses that flow from the *lower* electrode to ground. (As will be shown, this embodiment leads to serious errors.)
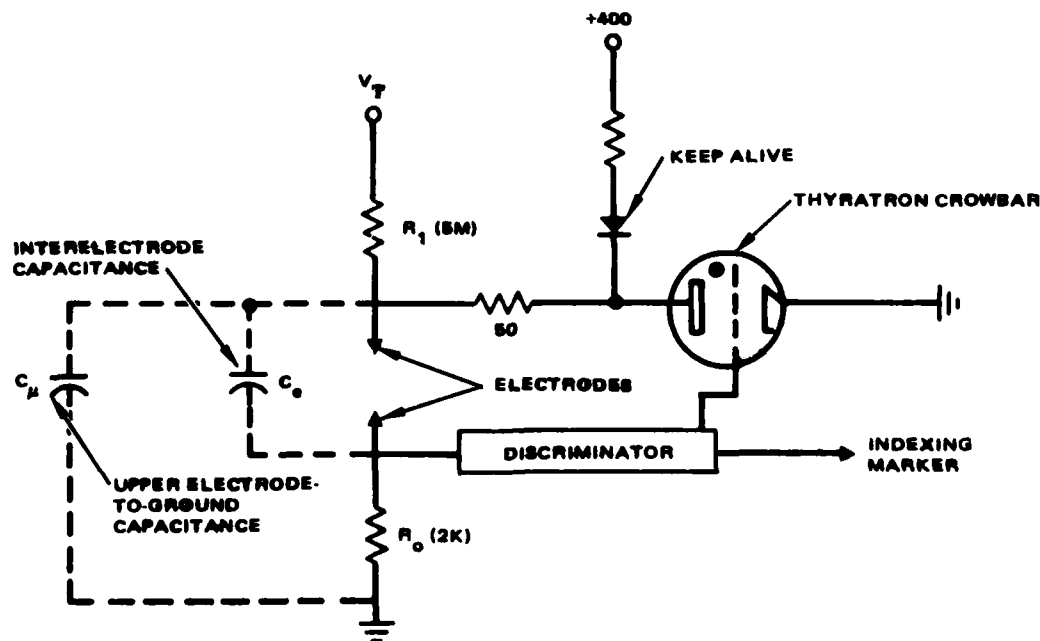

Figure 5-3. Nondestructive VBT.

Since the thyratron crowbar is *not* instantaneous, the success of the circuit in Figure 5-3 in providing the nondestructive VBT resides in the sample behavior itself. As shown in Figure 5-4, the question arises, can the sample dissipate the energy stored in the interelectrode

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capacitance C_e during the wait for the crowbar action without physical harm? (The upper electrode-to-ground capacitance, C_u, usually is negligible compared to C_e.) Initially the answer to this question would have been yes, but further diagnostic examination of films so tested gave such conflicting results that doubts arose as to whether nondestructive VBT was being observed when using the circuit of Figure 5-3. Indeed, the results obtained had been confounded by surface discharges. This confounding can best be understood by considering Figure 5-5, where a surface discharge is simply considered to short out capacitor C'_s. This shorting of capacitor C'_s leads to the waveforms shown in Figure 5-6.

Of particular interest in Figure 5-6 is the lower e_o waveform that closely mimics the expected results for a true breakdown, especially when various risetime problems are considered. Unfortunately, this waveform has been used as a measure of breakdown and to trigger the crowbar circuitry. It is obvious from Figure 5-6 that the true distinction between surface discharge and breakdown is best made by noting that in the former case the voltage e_u fails to go to zero.

To investigate breakdowns in which these surface discharges are absent, tests were performed with the electrode system shown in Figure 5-7. Here a fixed sample geometry is used with a conformable mercury electrode surrounded by a pool of mineral oil. This arrangement eventually became the standard small area test (SAT).

To minimize the interelectrode capacitance and thereby give nondestructive testing the best chance, the mercury contact with the sample was restricted to a 1 mm diameter circle ($C_e \sim 5$ pF). The test voltage was applied as a linearly rising ramp of 0.8 kV per second. (Here the desire was not to intentionally overvolt the sample test area.) In all cases, the samples failed abruptly, with e_u collapsing to zero in submicrosecond times. Subsequent examination of the failed areas invariably showed the presence of 10-60 μm holes. In addition, scanning electron microscopy showed that the borders of the holes were thickened, thus giving the distinct impression that the holes result from melting of the film with subsequent surface tension forces pulling the film into this pattern. Calculations of the electrical energy stored in the interelectrode capacitance of 5 pF show that sufficient energy exists under adiabatic conditions to cause such melting easily. Indeed, the whole breakdown act is so fast
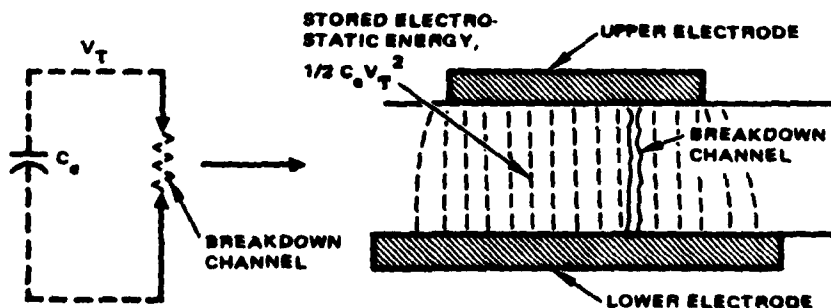
Figure 5-4. Energy dissipation in sample.
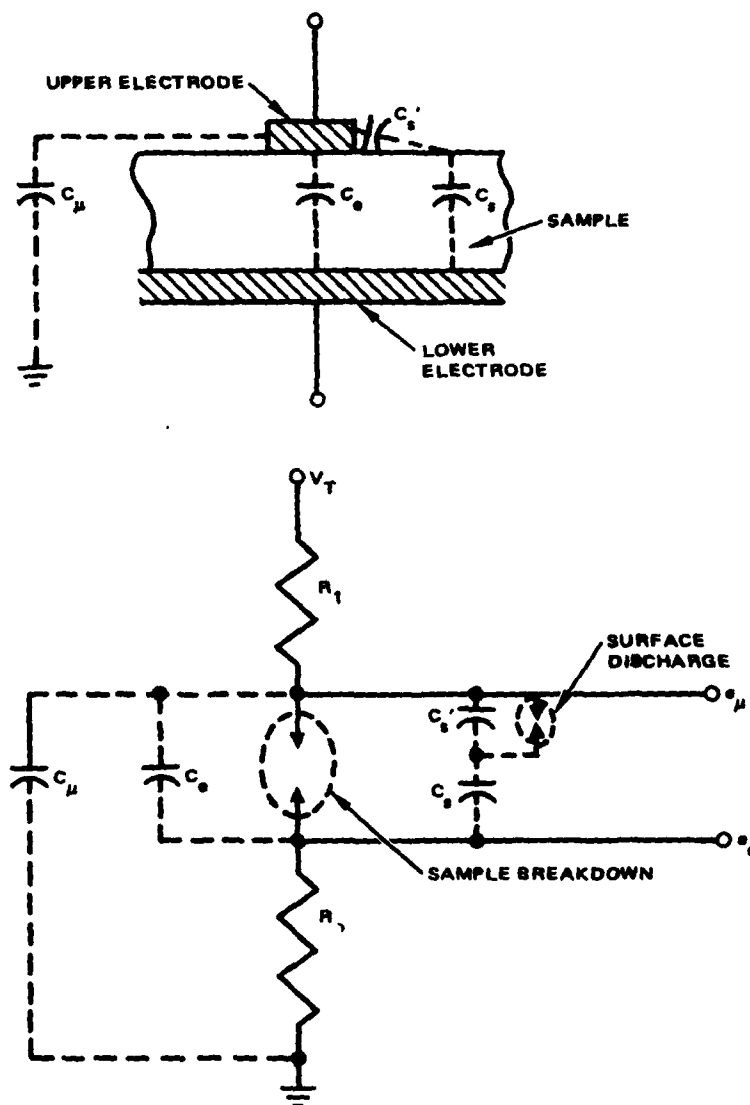
Figure 5-5. Electrode circuits.
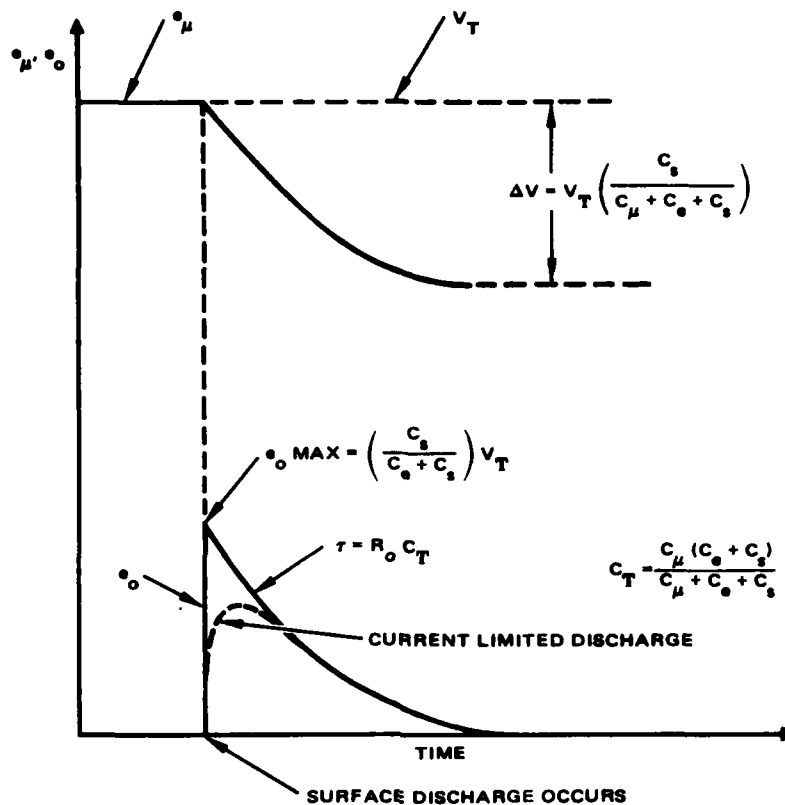
Figure 5-6. Waveforms.
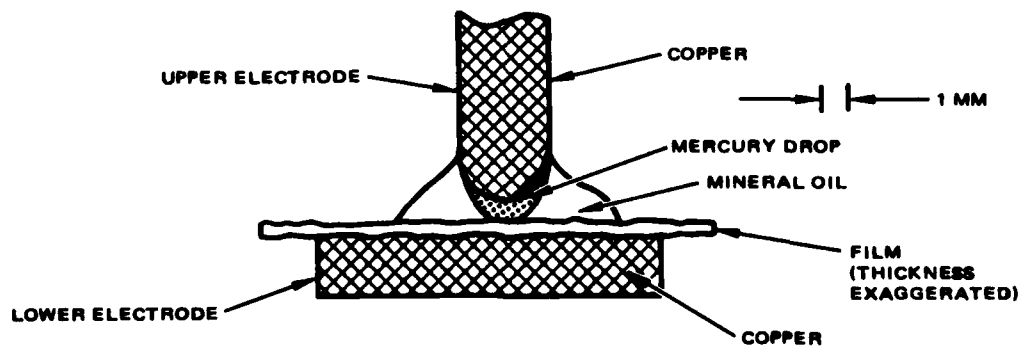
Figure 5-7. Surface discharge—free electrode system (SAT).

(nanoseconds) that no hope exists of performing nondestructive testing with electrodes of any reasonable dimensions.

Several reports in the literature claiming nondestructive VBT of dielectric films also show results that are confounded by the problem of surface discharges masquerading as true breakdowns.[*]

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[*]Riel, et al., "Z fur angew," Phys. 27, p. 261, 1969.

MEASUREMENT OF IONIC CONTAMINANTS IN POLYMER SOLUTIONS AND SOLVENTS

The dielectric loss of a polar liquid, such as methylene chloride, represents the sum of losses arising from dipole relaxing processes whose time scales intrude on the particular measurement frequency and losses representing DC conductivity. Since all polar liquids of *reasonable* viscosities have relaxation times of the order of 10^{-12} seconds, it is a very good approximation at low frequencies (10 kHz or lower) to ascribe all the observed loss to DC conductivity. Thus, if the familiar parallel model definition of dissipation factor is combined with the geometric definitions of capacitance and conductance, this important relationship may be defined:

$$D = \frac{2 \times 10^{12} g}{f \epsilon'}$$

where:

D = dissipation factor
f = frequency of measurement, Hz
ϵ' = dielectric constant of the liquid
g = specific conductance $(ohm \cdot cm)^{-1}$

This relationship indicates that the DC conductivity of a fluid may be measured by measuring its dissipation factor with a capacitance bridge connected to a simple capacitance cell containing the fluid as the dielectric (a small air capacitor submerged in the liquid will do). It is important to note the enormous numerical factor of 2×10^{12} relating D to g.

The conductivity of a liquid is solely ascribable to the presence of ions, which can arise from two sources. The first is the intrinsic property of the liquid to autoionize, e.g., $H_2O = H^+ + OH^-$. (As a practical matter, this autoionization is assumed to be negligible.) The second and more interesting source is the presence of adventitious contaminants that can dissociate in the dielectric liquid to yield ions. Here the enormous influence that the dielectric constant of the liquid plays on the extent of this dissociation must be understood. As shown in Figure 5-8, a few parts per million of a quaternary ammonium salt in mineral oil ($\epsilon' = 2.2$) would exist almost completely in the form of neutral ion pairs, $[R_4N^+NO_3]^0$, and as such would make little or no contribution to the liquid's dissipation factor. However, if this same concentration of this salt were present in methylene chloride ($\epsilon' \sim 9$), its contribution to the liquid's dissipation factor would be enormous because the dissociation constant of the salt has been increased by a factor of 10^{12}! (It is reasonable to infer that the often observed fact that liquids of high dielectric constant are always quite lossy arises from this exponential influence of dielectric constant and not on a particularly enhanced concentration of impurities over that present in liquids of lower dielectric constant.)

Thus, the basis is attained of an extremely sensitive and simple testing system for assaying the concentration of ionic contaminants in polysulfone resin, solutions and solvent systems;
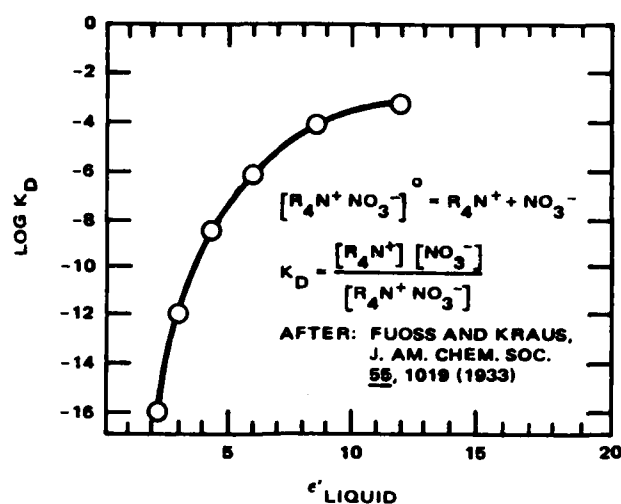
Figure 5-8. Ion pair dissociation versus
liquid dielectric constant.

i.e., known quantities of these materials are dissolved in carefully purified (distillation) methylene chloride and the dissipation factor of resulting solution measured.

POLYMER FILM CONDUCTIVITY

All measurements were performed with gold sputtered electrodes, which were offset from one another so that limp, aluminum foil electrodes could be attached with Aquadag. The effective area was calculated from capacitance measurements.

FILTRATION STUDIES

Many experiments were performed to evaluate the effect of filtration of the casting solution on the breakdown properties of polysulfone film. In some cases, before the films were actually cast, the performance of the filter was evaluated by diluting the casting solution with filtered methylene chloride and filtering the solution on Millipore filter pads and counting the number of particles. A typical analysis of this type is presented in Table 5-1.

TABLE 5-1. PARTICLE ANALYSES OF FILTERED
CASTING SOLUTIONS

Filter Type	Micrometer Rating	Particle Analysis (per gram resin)	
		7-15 μm	15 μm
Cox	1	6.1	2.1
Cox	2	5.7	1.0
Cox	5	17.0	5.2
Cuno 10A	10	3.7	1.8

Films prepared from filtered casting solutions were cast on the laboratory caster and the number of breakdowns counted using a large area test (LAT). Breakdown counts per unit area are shown in Figure 5-9 as a function of stress.
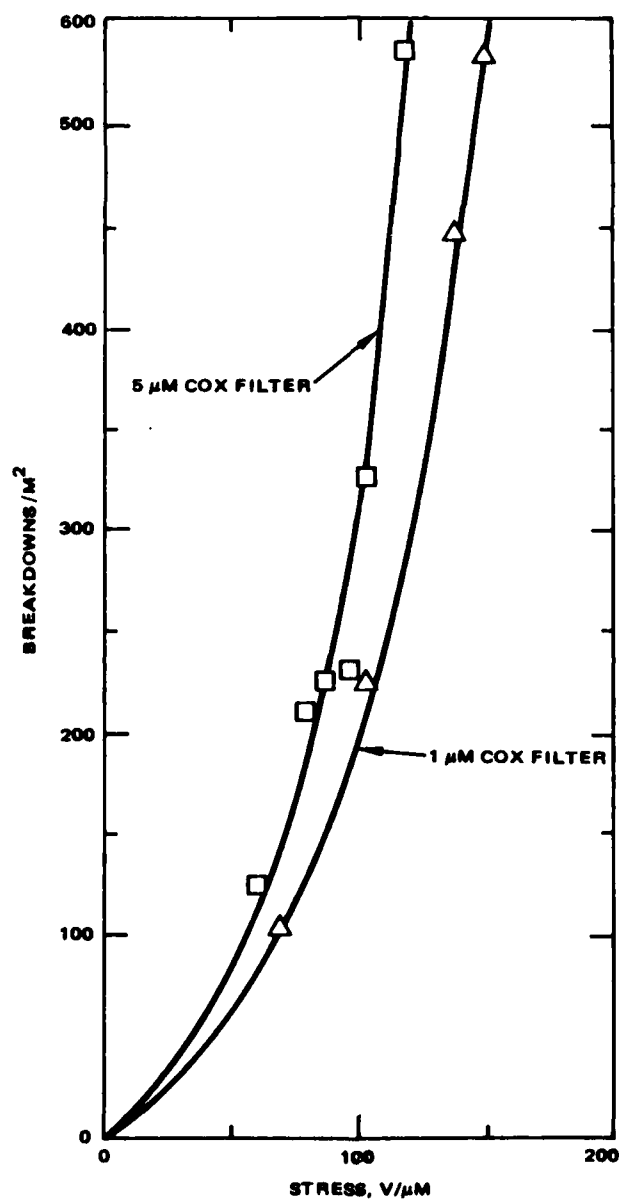
Figure 5-9. Breakdowns versus voltage stress.

In an attempt to improve the breakdown performance of the laboratory cast films, various other procedures were employed to eliminate particulate contamination during the casting operation. These steps included a second stage of filtration for the drying air for the lab caster and enclosing the caster in a tent to reduce airborne contamination. These additional procedures did not affect the occurrence of dielectric breakdown.

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As these experiments proceeded, it became increasingly apparent that mere filtration of the casting solution and attention to the details of maintaining a particle-free film were not the whole reason for breakdown at the voltages of interest. This fact is noted by considering the particle counts presented in Table 5-1 and the breakdown results in Figure 5-9. Consideration, for example, of the particle counts for the 1 μm Cox filter shows that there are a total of 140 particles greater than 7 μm in a square meter of film such as that presented in the breakdown results of Figure 5-9. Since the film is about 14 μm thick, there are not nearly enough particles to account for the large number of breakdowns observed.

In light of the above experiments, other mechanisms for dielectric breakdown were considered. In a series of experiments that are discussed in the next section, the film samples were found to contain mobile charge carriers, probably dissolved ionic impurities, which move through the dielectric when voltage is applied and cause distortion and enhancement of the electric field.

CHARGE MOBILITY EXPERIMENTS

Small Area Tests

To avoid the influence of conducting particles on dielectric breakdown, the small area test (SAT) was employed. This device examines only a few square millimeters of film and is described more fully in the Test Development Section of this report. In this test, a ramp of 5 kV per second is applied to the sample until breakdown occurs. A storage oscilloscope records the voltage.

In a crucial experiment, a bias voltage of 1000 V (a fraction of the ultimate breakdown voltage) was applied to the film for various times before starting the voltage ramp. The results, as shown in Figure 5-10, show that the longer the bias voltage is applied, the higher the ultimate breakdown voltage.
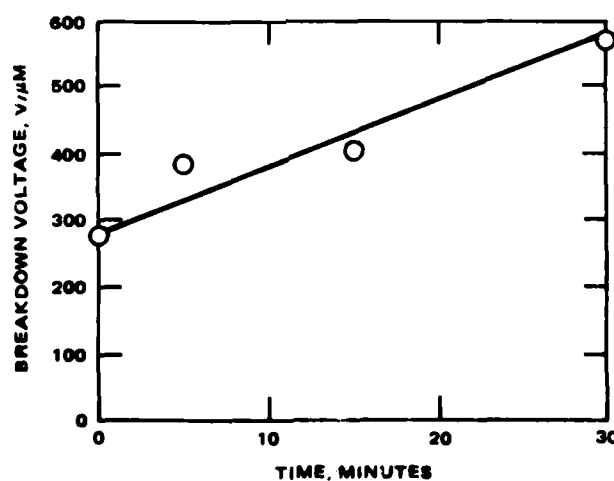
Figure 5-10. Breakdown voltage stress versus time of applied bias voltage.

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Moreover, in these tests if the bias is removed for a time equal to its application before the ramp is started, the breakdown voltage returns to its original value. From these experiments, it is evident that some migration of charge carriers within the dielectric must be occurring to account for the difference in breakdown voltage. This idea is especially cogent since films such as polypropylene, which have an initially high breakdown voltage by this test, do not display a higher voltage when "polarized" as described above. This observation suggests that when a few mobile charge carriers are present, application of a bias voltage will have little effect on the breakdown voltage.

While the interpretation of these results is somewhat speculative, a possible explanation exists in the field distortion illustrated in Figure 5-11. In Figure 5-11a, the sample is completely non-polarized, and the charge carriers are uniformly distributed throughout the dielectric. In Figure 5-11b, a polarizing voltage has been applied for some time causing a field distortion because of the presence of charge carriers adjacent to the electrodes. When a ramped voltage is applied to this situation, the electric field (the slope of the curve in Figure 5-11b) will be small in the bulk of the sample but very large in a very thin region adjacent to the electrodes. It is well known for most dielectrics that very thin layers of insulating material display extremely high dielectric strength. Furthermore, an intermediate condition might exist wherein the field would be high near the electrodes but not in a layer thin enough to display high breakdown voltage. Such a situation is indeed observed as discussed next.
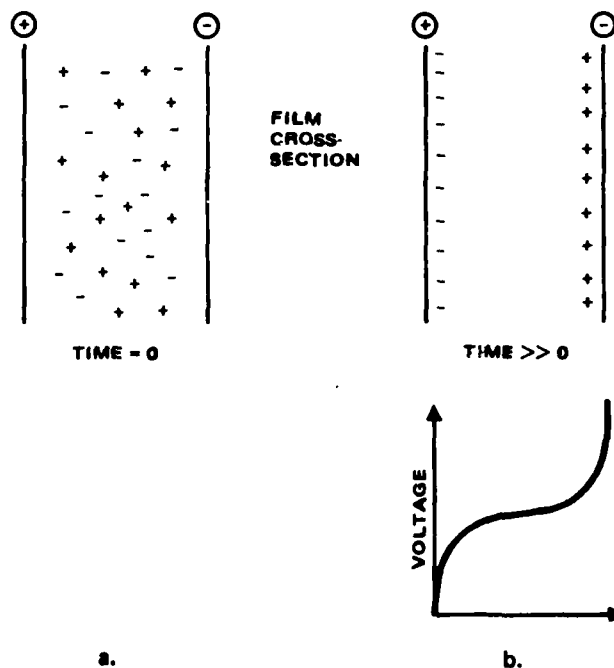
Figure 5-11. Charge distribution within
"polarized" dielectric.

Time-Dependent Breakdown

In a typical breakdown experiment, when voltage is applied to a sample the dielectric weak spots are expected to fail and thereafter breakdowns will cease. Application of a higher voltage will cause more weak spots to fail and so on. Such is not the case with many of the films tested in this work; instead breakdowns continue as voltage is left on the sample.

A revealing experiment was conducted with polysulfone film using the small area breakdown testing apparatus discussed in the previous subsection. In this experiment, the average breakdown voltage under high ramp rate conditions was determined for the sample. Then, using the same setup, a percentage of that voltage was applied under DC conditions until breakdown occurred. The results are shown in Figure 5-12.
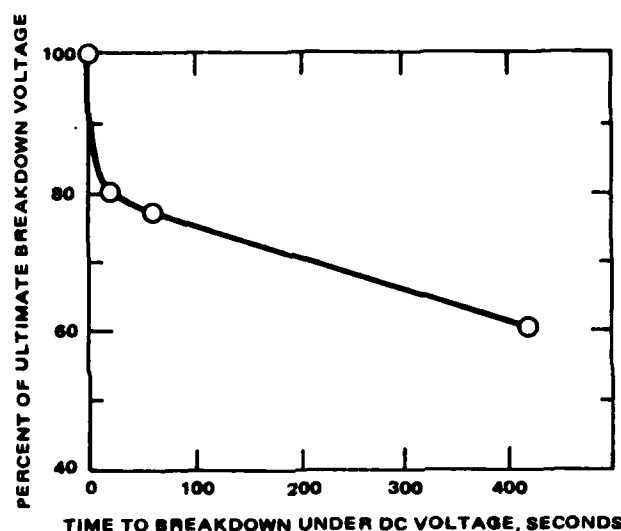
Figure 5-12. Percent of ultimate breakdown voltage under ramp conditions versus time of application of DC voltage small area test.

These results show that as the DC voltage is reduced to some value less than the high-ramp rate breakdown voltage, breakdown still occurs but at increasingly longer times. These results can be interpreted as indicating that a critical field distortion pattern can be created by the mobile charge carriers. Thus, it can be argued that the field is much enhanced near the electrodes but over dimensions such that the sample fails first near the electrodes and then in the interior of the dielectric. The progression in time of the voltage within the sample is illustrated in Figure 5-13.

In another experiment in which time dependent breakdown was observed, a constant DC voltage was applied to a large area of commercial polysulfone film using the metallized film/ brass plate electrode arrangement. The results of this experiment are presented in Figure 5-14. As shown, the cumulative breakdown counts increase indefinitely with time. In fact, such experiments have been continued for as long as 30 minutes with no sign of
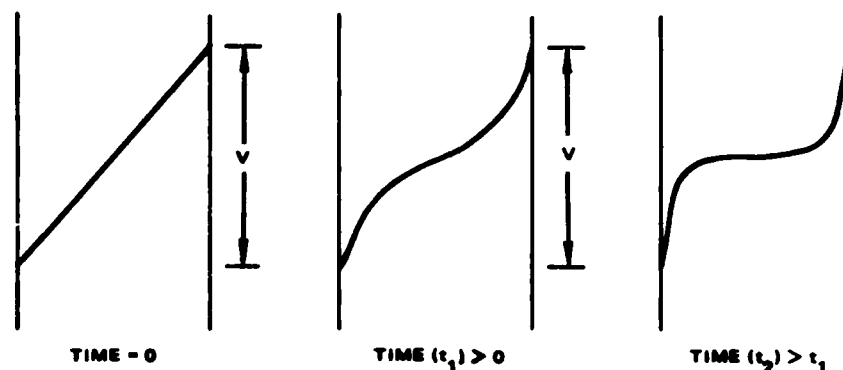
Figure 5-13. Field distortion versus time.
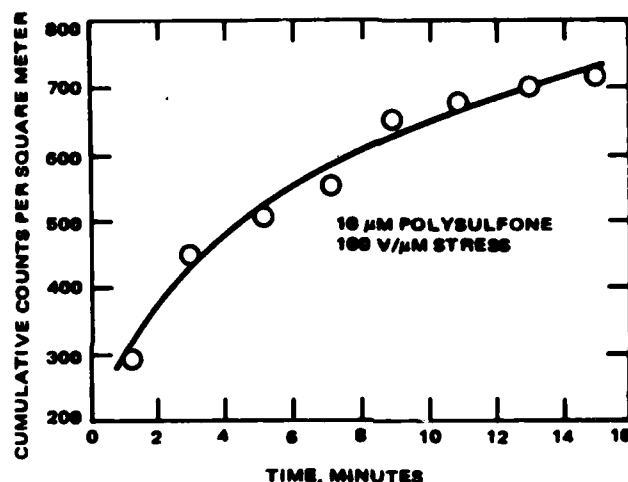
Figure 5-14. Time-dependent breakdown cumulative
counts/meter^3 versus time of application of voltage.

stopping. In such a test, it is reasonable to ask why one spot breaks down before another when the same voltage is applied to the entire sample. Evidently, the mobile carriers do not build up uniformly at the electrodes due to slight differences in carrier concentration or mobility.

Some interesting comparative experiments were performed with other dielectric films to determine if they also exhibit time-dependent breakdown. Using the same metallized film/ brass plate electrode arrangement as above, both polyethylene terephthalate (Mylar) and polypropylene films were tested. At the modest voltages available with this equipment, no time-dependent breakdowns were observed in the polypropylene samples but were observed in the Mylar samples as indicated in Figure 5-15. Thus, while the breakdown voltages may not be the same, time-dependent breakdowns are not restricted to polysulfone films.
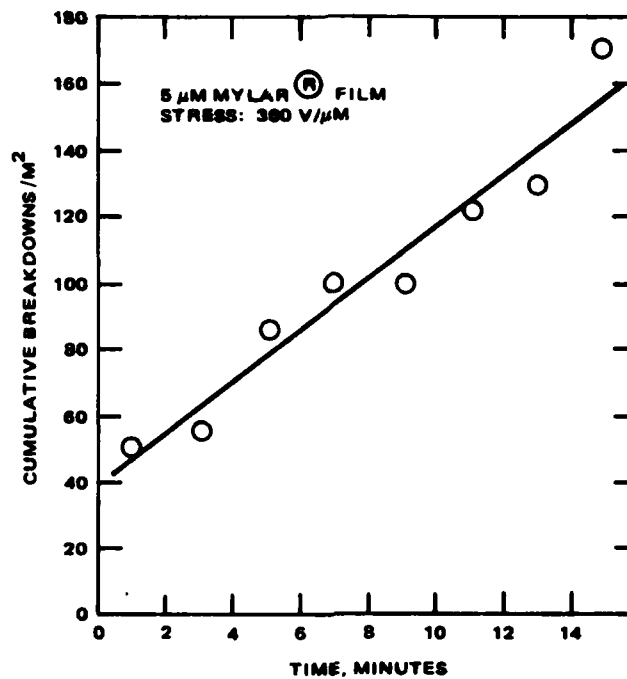
Figure 5-15. Cumulative breakdowns versus time
of application of voltage.

Film Conductivity

To assess the changes in conductivity of the film samples because of the movement of charge carriers, a cell was constructed to measure film conductivity at various voltages, temperatures and times. The time-dependence of these measurements is particularly interesting in light of the results discussed above concerning time-dependence of dielectric breakdown. Current density versus time is shown in Figure 5-16 for a commercial sample of polysulfone film with an applied voltage of 1000 volts. From these data it is clear that the current is initially high but decreases with time, presumably as the mobile charge carriers accumulate near the electrodes. After 15 minutes the polarity was reversed, and the current was observed again to be initially high but to decrease with time and at approximately the same rate as initially. In these experiments, current density rather than a computed resistivity has been plotted as a function of time because of the non-ohmic nature of the conductivity. This behavior is best illustrated in Figure 5-17 in which current density is plotted as a function of voltage stress for the same sample as in Figure 5-16. In both figures, the sample area used to determine the current density was determined from capacitance measurements. (In calculating the current density, the current is assumed to be flowing uniformly through the sample which may not be the case.)

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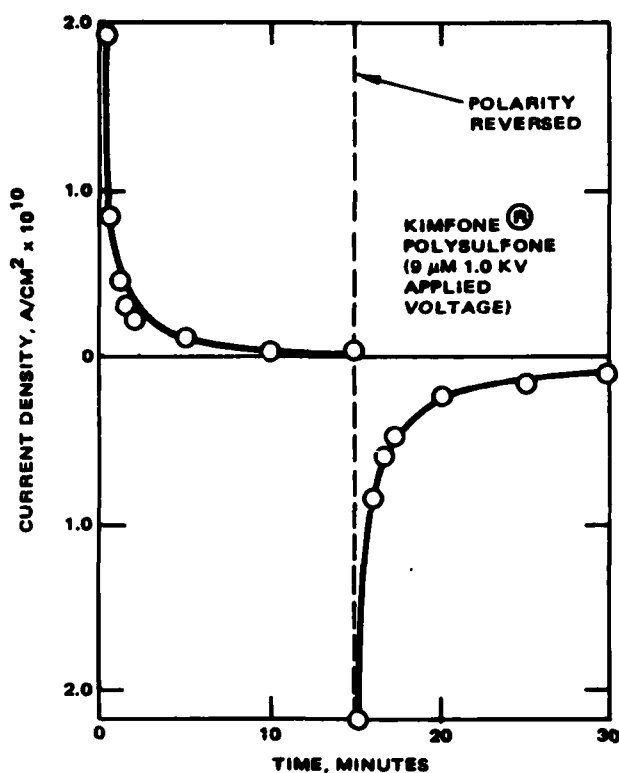
Figure 5-16. Current density versus time for commercial polysulfone film.
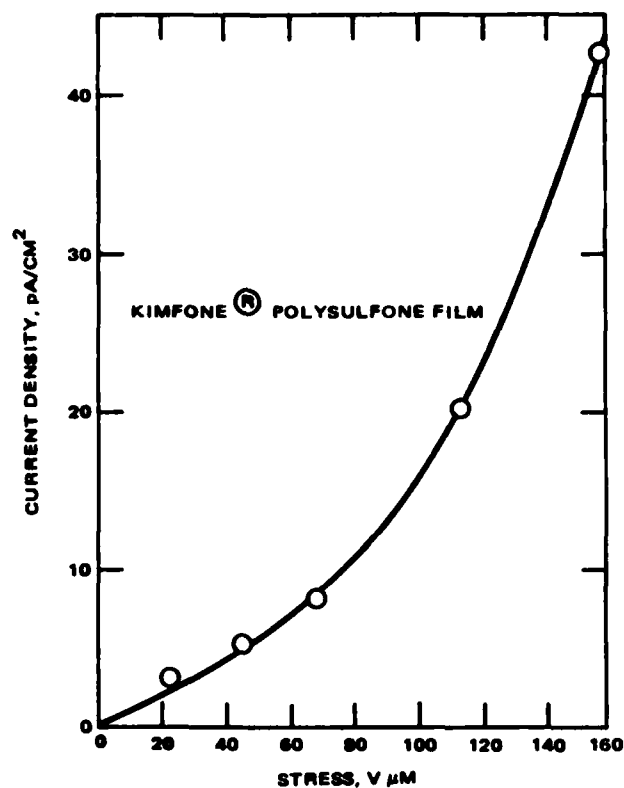
Figure 5-17. Current density versus voltage stress for commercial polysulfone film.

DECONTAMINATION PROCEDURES

From the experiments discussed above, it is clear that some procedure must be developed to remove or ameliorate the effect of the ionic impurities present in the film. Further, some test must be available to assess the efficacy of the decontamination procedure. Accordingly, many procedures were tried for decontaminating the casting solutions. Since the composition of the contaminants was unknown, the procedures selected generally were applicable for a wide variety of organic and inorganic materials. The evaluation included absorption with activated carbon, extraction with deionized water, absorption with silica gel, activated alumina, talc, and diatomaceous earth. Another procedure employed was filtration of the polymer solution through a thick pad of chemically pure cellulose fibers such as used for manufacture of kraft capacitor tissue. Still further purification was attempted by exhaustively extracting contaminants from the cast film with methanol in a Soxhlet extractor.

The procedure used in purification was to first dilute the normally very viscous casting solution with distilled methylene chloride to provide a viscosity low enough to get good mixing of the absorbent and the polymer solution. Then the absorbent was added to the

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solution, usually at a level of about 50 percent of the resin weight, and shaken for 16 hours. The slurry was then filtered to remove the absorbent and concentrated by evaporation of methylene chloride using a rotary evaporator.

The technique for evaluating the effectiveness of the decontamination procedure was to measure the dissipation factor of a dilute methylene chloride solution of the polymer. This procedure (described more fully under Test Development) is performed rather simply by dipping a clean air capacitor into the polymer solution. The results must be interpreted carefully, however, since, regardless of the nature of the ionic impurity, they all behave as weak electrolytes when in a solvent with a low dielectric constant such as methylene chloride. The extent of the dissociation of the ion pairs is then a function of the concentration of the electrolyte and follows the classical Ostwald dilution law. Unfortunately, the concentration of the impurities is not known; therefore, the dissipation factor results only can be compared at equal polymer concentration. The relative extent of the ionic contamination can be determined because of the ability of the polymer to dissolve in a convenient solvent. Such determinations would be much more difficult in a polymer such as polypropylene since it does not have a convenient solvent.

From the tests and procedures described above it was apparent that sorption of the ionic contaminants was most effectively done with activated carbon. A number of determinations of dissipation factor for various samples are compiled in Table 5-2. From tests 1 and 2 in Table 5-2, it is clear that both the technical grade methylene chloride normally used to make casting solution and the polysulfone resin are replete with ionic contaminants. Test 3 indicates that distillation does a reasonable job of removing ionic contaminants. Commercial polysulfone film samples showed a considerable reduction in ionic contamination relative to the resin as shown in tests 4 and 5 but not nearly as great a reduction as achieved when the polymer solution was treated with activated carbon (test 6). The reduced amount of ionic contamination in the commercial films compared to the resin may be caused by sorption by the filtration medium normally used for filtering casting solutions. These filters are typically cellulosic in nature, and cellulose has been shown to provide some reduction in ionic content (test 7).

Many films prepared from casting solutions treated to remove ionic contaminants were tested using the small area tester (SAT) described previously. Since the data from these determinations contained considerable scatter, at least 10 separate readings were made for each film and the standard deviation computed. A number of these determinations are given in Table 5-3 with the various treatments applied to the casting solutions. From these data and others not listed, the treatments seem to have some influence on the small area test results. However, the scatter in the data make such comparisons difficult.

Results of conductivity measurements on treated and untreated films show that some reduction in conductivity can be obtained. The curves in Figure 5-18 show current density versus voltage stress for two films with the same thermal treatment before testing. The

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TABLE 5-2. DISSIPATION FACTOR MEASUREMENTS ON VARIOUS SOLUTIONS

Test	Sample Designation	Percent Resin Weight	Percent DF, (1 kHz)
1	Technical Grade Methylene Chloride	0	>100
2	Polysulfone Resin (as received)	0.65	>100
3	Distilled Methylene Chloride	0	5-8
4	Commercial Polysulfone Film Sample—good SAT	1	38
5	Commercial Polysulfone Film medium SAT	1	58
6	Polysulfone Solution Treated with Activated Carbon (50 percent of resin weight)	7	20-40
7	Polysulfone Solution Treated with Cellulose	1	73

TABLE 5-3. SAT BREAKDOWN FOR TREATED FILMS

Test	Treatment	Mean Breakdown Voltage, V/μm	Standard Deviation, V/μm
1	No treatment—Lab Cast Film	400-450	100-150
2	Cellulose	610	54
3	Water Extraction	520	83
4	Activated Carbon	620	90
5	Diatomaceous Earth	550	60

untreated film was cast from a solution that went through the same procedure as the treated solution except that the activated carbon was omitted. The results show that not only is the conductivity generally lower with the treated film but also more nearly ohmic.

While experiments measuring the ionic contamination in the casting solution, small area tests, and film conductivity all show some improvement in film properties, these improvements were a result of decontamination procedures and were not carried through to large area breakdown tests or time-dependent breakdown. To evaluate the dielectric strength properties of films without the necessity of handling or further contaminating the film, the laboratory casting machine was fitted with an aqueous line contact electrode system as described in the Test Development Section.

A polysulfone solution was treated with activated carbon and filtered through a composite filter medium consisting of a 5 and 0.65 μm filter. The dissipation factor of the solution thus
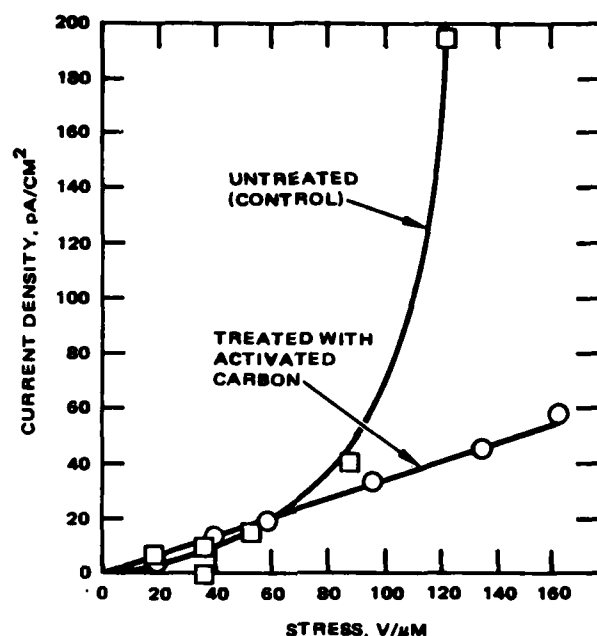
Figure 5-18. Current density versus voltage stress for
treated and untreated film.

prepared was 43 percent in a 7 percent polymer solution. The breakdown counts using the
aqueous electrode system are shown in Figure 5-19. Comparison of this figure with Figure 5-9 shows that very little improvement has been gained in large area testing.

Films cast from treated solutions also exhibit time-dependent breakdown. For these
experiments, a number of breakdown tests were performed using film samples of approximately 6 cm diameter that were sputtered with gold on both sides, in addition to large area
breakdown tests using the brass plate/metallized film electrodes. These extra precautions
were taken to avoid any potential problems with air gaps between the sample and electrodes.

POLYIMIDE FILM

During this work, a new polyetherimide resin was commercialized by the General Electric
Company. This new material, with the tradename of Ultem® has many properties to recommend it for high energy density capacitors such as proposed in this study. The relevant
thermal, mechanical and dielectric properties of both polysulfone and Ultem® are given in
Table 5-4.

From these properties, the permissible operating temperature of capacitors made from
Ultem® film is assumed to be higher than for polysulfone. In addition, because of its superior
strength, fabrication of capacitors may be easier. Finally, because of the very high oxygen
index for this material, the flammability of capacitors is considerably reduced.
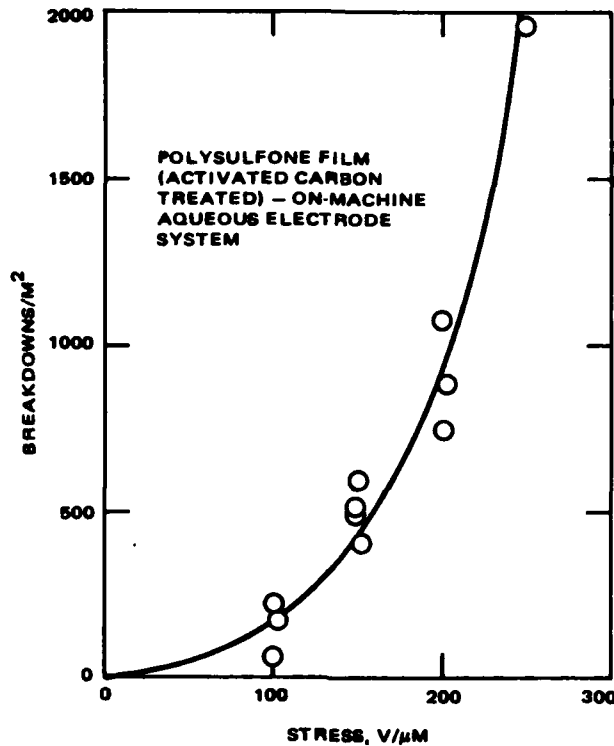
Figure 5-19. Large area breakdown versus voltage stress.

TABLE 5-4. PROPERTIES OF ULTEM® AND POLYSULFONE RESIN

Property	Polysulfone	Ultem®
Glass Transition Temperature, °C	190	217
Ultimate Tensile Strength, psi	15,000	26,000
Oxygen Index (bulk resin)	30	47
Dielectric Constant 23°C, 60 Hz	3.07	3.0
Dissipation Factor 23°C, 60 Hz	0.0008	0.0012

Accordingly, film was cast from this material, stretched in the fashion as for polysulfone (albeit at a higher temperature) and slit into bobbins. The breakdown characteristics of the film were determined using both the aqueous electrode and the metallized film electrode systems. The results of these tests are shown in Figure 5-20. When this curve is compared with those for polysulfone, considerable improvement in breakdown characterisitic is noted over polysulfone. Therefore, Hughes selected this material for further study and evaluation.

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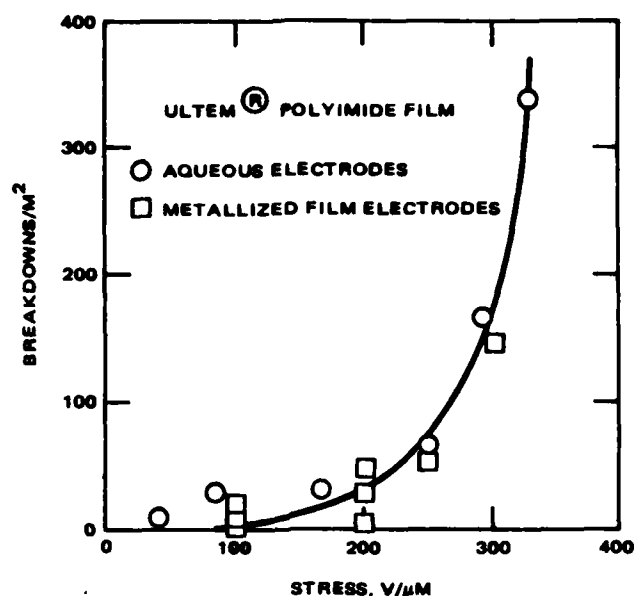
Figure 5-20. Large area breakdown counts/meter^2 versus voltage stress Ultem® polyimide film.

TEXTURED FILM

At the outset of this work, one stated goal of the program was to develop a technique for providing a film with a textured surface. It was thought that such a material might provide a capacitor winding that could be impregnated and thus avoid the necessity of using kraft capacitor tissue with its inherent moisture absorption. To fulfill this need, Ultem® polyimide film was prepared on a sandblasted laboratory casting drum. Scanning electron micrographs of this film and a comparative micrograph of film cast on a polished casting drum are shown in Figure 5-21. These micrographs show that, while the exact extent and nature of the roughness introduced in this experiment may not be optimum, casting on roughened drums does seem to be a viable technique for producing textured film.

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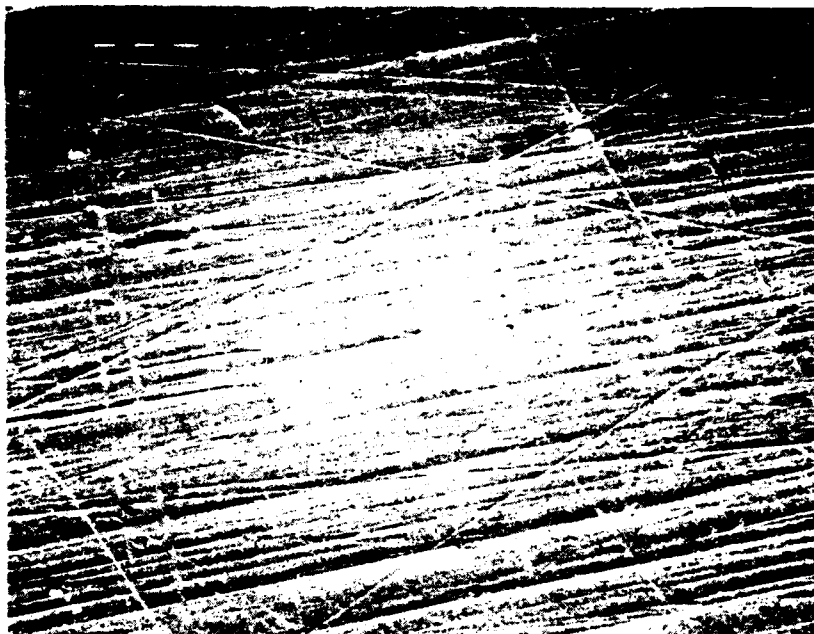
a. Smooth Surface
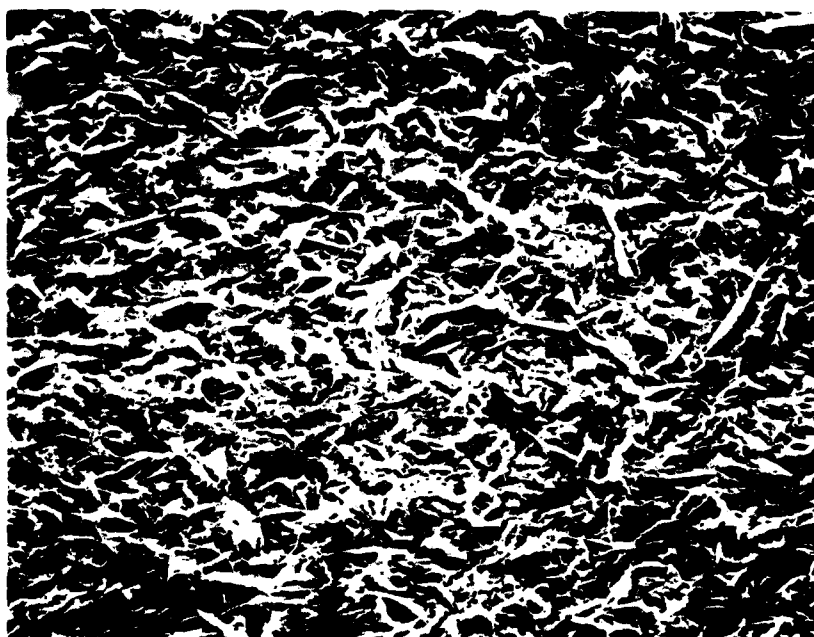
b. Textured Surface

Figure 5-21. Scanning electron micrographs of polyimide film (250X).

6.0 TASK III—FINAL SELECTION

RECOMMENDED DIELECTRIC SYSTEMS AND REVISED ESTIMATES

Based on the results of the work of Schweitzer, the dielectric systems have been revised to include the solution-cast polyimide in cases where it shows an advantage over polysulfone. Because the development resulted in a material of higher temperature capacity rather than of improved dielectric strength, the capacitors in general would be expected to have the same or slightly better energy density, but they could be operated at higher cold-plate temperature or higher duty cycle. Thus, the overall system weight would be lower even if the energy density measured in the laboratory shows limited improvement.

High Repetition Rate Capacitor

In this design, similar to design B on page 86 of AFWAL–TR–80–2037, the operating field has been reduced slightly. However, the parts should have better life and better power throughput than the components made from polysulfone.

Number of dielectric layers	: 5
Polyimide layers	: 2.6μ (24 gauge)
Kraft layers	: 3/10.1μ (0.4 mil)
Foil	: Aluminum /6μ
Dielectric fluid	: Dioctylphthalate

Estimates are:

Average electric field	: 4464 V/mil
Average dielectric constant	: 4.302
Energy density, complete pad	: 0.22 J/g (99.8 J/lb)

Low Repetition Rate Capacitor

This component has the same pad design as the high rate component, but a slightly higher operating field.

Number of dielectric layers	: 5
Polyimide layers	: 2/6μ
Kraft layers	: 3/10.1μ
Foil	: Aluminum, 6μ
Dielectric fluid	: Dioctylphthalate

Estimates are:

Average electric field	:	4762 V/mil
Average dielectric constant	:	4.302
Energy density, complete pad	:	0.25 J/g (113.6 J/lb)

DC Capacitor

For this design, either the design for the pulse capacitor as above or the previously cited design using PVDF can be used. During the second phase, the PVDF component will be fabricated if the material is available. However, a system would be simplified if the same component could be used for both functions.

High Frequency AC Capacitor

The polyimide material has particular application here, as this component will unavoidably get very warm.

Number of dielectric layers	:	2
Polyimide thickness	:	6μ (0.24 mil)
Foil	:	Aluminum, 6μ
Dielectric fluid	:	Silicone

Estimates for these components are:

Average electric field	:	3125 V/mil
Dielectric constant	:	3.2
Energy density, complete pad	:	0.08 J/g (37 J/lb)

If the textured material were available, it would be interesting to construct a component with two layers of 5μ film (20 gauge); then 50 J/lb with a field of 3750 V/mil would be attained.

7.0 CONCLUSIONS AND RECOMMENDATIONS

During this work, a great deal has been learned about the nature of dielectric films in general and polysulfone film in particular. At the outset it was thought that a superior dielectric film could be produced by merely eliminating as much of the particulate contamination as possible. Relatively little attention was given to the dissolved ionic impurities present in the film, which have been shown to play a significant role in dielectric breakdown. It was shown that these contaminants enter the films via the resin and the solvent and can be removed, at least partially, by appropriate treatment of the polymer solution. Unfortunately, to date these treatments have not been developed sufficiently to significantly improve the breakdown characteristics in several key tests. Hughes is especially concerned about time-dependent breakdown and believes that it occurs because of migration of the ionic impurities and the accompanying field distortion. The remaining problems, therefore, are to determine to what extent the impurities must be removed and to develop a practical technique for doing it. The development of dielectric films of *substantially* higher energy density than those presently available must await the solution to this problem.

The technique of casting films on a roughened casting drum has been shown to be useful in preparing textured films. Further work is necessary in this area to better define the extent and nature of the roughness required to make an impregnable capacitor. This work is the first practical demonstration of a general process for making films of many types that may be used in high-voltage kraft-paper-free capacitors. The elimination of kraft paper will allow a higher operating temperature, lower dissipation factor, higher average field, and generally improved assembly processes.

This work has also led Hughes to propose a new material for use in high energy density capacitors. While many of the same problems of particulate and ionic contamination must be considered for this material, tests have shown some extremely promising results. Further testing on capacitors will be necessary to develop the processing conditions necessary for this material and will be completed in the second half of this program.

This new material, a General-Electric-developed polyimide, may well become the universal material for many military capacitor applications. This material has a higher temperature capability than any other film material except DuPont Kapton, but since it is solution-castable it can be made at any thickness desired with very high film quality. Its electrical properties are very stable with temperature and frequency. It is easy to solution-cast, has a high ultimate tensile strength, and has generally excellent mechanical properties. Its dissipation factor is 50 percent larger than polysulfone (at 1 kHz, R.T.), but is still only about a third that of Mylar.

In the proposal the most promising existing material candidates were identified for development. It was estimated that by reducing the frequency of electrical imperfections

polysulfone film could operate at electrical stresses significantly above those currently employed. An improvement in energy density was projected and preliminary pad designs, utilizing polysulfone film and kraft paper, were given. These designs were similar to designs previously developed.

Based on the results of the work of Schweitzer, the dielectric systems have been revised to include solution-cast polyimide where it shows an advantage over polysulfone. Because the development resulted in a material of higher temperature capacity, rather than of improved dielectric strength, the capacitors would in general be expected to have the same or slightly better energy density. They could be operated at higher cold-plate temperature or higher duty cycle and the overall system weight would be lower.

A comprehensive Material Test Plan was prepared. The plan is easy to implement. It is intended primarily for the use of people developing specific sets of materials. It could easily be used to produce a military specification which could be used for materials in their final form.

The plan is divided into three groups of materials: films, oils, and papers. Test procedures are listed by group. Most tests are performed per ASTM specifications, with special tests explained. Test requirements are summarized in tables.

APPENDIX A

STATEMENT OF WORK

ADVANCED CAPACITORS

1.0 OBJECTIVE

The objective of this effort is the development of dielectric materials required for the lightweight, high energy density capacitors needed for high power, airborne power systems.

2.0 SCOPE

2.1 This program shall include the modification of existing dielectric materials and/or the development of new materials as required to meet the capacitor performance requirements as specified in section 4.6. The effort shall involve the fabrication and testing of small samples of selected dielectric materials as well as the fabrication and testing of capacitor pads employing the improved dielectric materials.

2.2 A capacitor pad shall be defined as an element of capacitor construction consisting, typically, of a rolled winding of conducting foils separated by the dielectric medium. In a given capacitor, several pads may be connected in series/ parallel configurations to obtain the desired characteristics. However, capacitors consisting of single pads are also possible.

2.3 Four classes of capacitors shall be considered. These are: (1) High rep rate pulse capacitors, (2) Low rep rate pulse capacitors, (3) High voltage filter capacitors, and (4) High frequency AC capacitors. Classes (1) and (2) refer to capacitors used in pulse forming network (PFN) applications while class (4) refers to their use in series resonant inverters.

3.0 BACKGROUND

3.1 Having identified capacitors as one of the heavier components of an airborne power conditioning system, the Air Force Aero Propulsion Laboratory (AFAPL) has, for the past several years, been engaged in programs aimed at increasing capacitor energy density. The most recent of these (contract F33615-75-C-2021, Hughes Aircraft, Culver City CA) has, through improved design and fabrication techniques, pushed existing dielectric materials to the limit of their electrical and thermal capabilities. An interim report ("Capacitors for Aircraft High Power," AFAPL-TR-77-40, September 1977) describes a portion of this effort.

3.2 The Hughes program has developed capacitor technology to the point where any further improvements are limited by the dielectric materials of which the capacitors are made. However, this limit is not necessarily one imposed by the intrinsic properties of the materials themselves, but rather by those values at which they can be realistically and reliably operated in an actual capacitor environment. Therefore, improvements or modifications to these materials to allow operation at or near these intrinsic values, or development of entirely new materials are required to further increase capacitor energy density.

3.3 The capacitors of interest to the AFAPL typically are of a multi-layer sandwich construction consisting of several layers of plastic dielectric film each separated by a layer of Kraft paper. The two outermost layers are metallic foils which serve as the conducting plates of the capacitor. This sandwich is then wound on a mandrel to obtain the required capacitance and impregnated with a dielectric fluid.

3.4 The multi-layer construction is necessitated by the requirement for high voltage (e.g., tens of kilovolts) coupled with the imperfections (e.g., pinholes and thickness variations) of the plastic dielectric films that are presently available. Paper is usually required as a wicking agent to insure complete impregnation by the fluid and elimination of any voids between film layers which may lead to corona and eventual capacitor failure.

3.5 Any irregularities occurring in the capacitor materials (e.g., wrinkes, folds, irregular edges, etc.) either introduced by the capacitor fabrication process or as a result of the manufacture of the various materials themselves, result in field enhancement. This, in turn, limits the electrical stress (V/mil) that can be applied to the bulk dielectric and, therefore, the voltage at which the capacitor can operate.

3.6 If plastic films can be developed which have high dielectric strength, and/or no imperfections, a given capacitor would require fewer layers of dielectric material (all else being equal), which results in reduced weight and volume. If this film was also designed such that paper was not required for complete impregnation, still further gains could be made. Ideally, the dielectric film would be such that only one layer would be required, possibly eliminating the need for both paper and impregnant.

3.7 However, capacitor improvements in terms of energy density (joules/pound) can be realized through development of dielectric systems with high dielectric constants ($>$ 10), low dissipation factors ($<$ .01%), high operational dielectric strength ($\geq$ 5kv/mil), long life, and which are pinhole free, of uniform thickness, non-flammable, non-toxic, and will perform over the military temperature range. Materials presently exist which exhibit these properties individually but not collectively. In addition, many of these properties are frequency and/or temperature dependent and, quite possibly, no one material can be developed which exhibits all the desired characteristics over the range of Air Force requirements. Tradeoffs will have to be made. However, if the process of designing dielectric materials is well established, it should be possible to design dielectric systems to fit the application.

4.0 TECHNICAL REQUIREMENTS

   4.1 PHASE I: MATERIAL TESTING AND SELECTION

      4.1.1 Task I: Preliminary Material/Dielectric Systems Selection

         4.1.1.1   The contractor shall identify promising existing material candidates for modification and/or recommend generic types of materials for development. These recommendations shall be based on an analysis of the material requirements necessary for dielectric systems satisfying the capacitor requirements as specified in section 4.3.

         4.1.1.2 · The contractor shall make preliminary estimates as to the expected improvements in specific dielectric properties through his recommended material modification and/or development. These estimates shall be supported by technical discussions indicating how they were derived.

         4.1.1.3 The contractor shall consider material properties such as dielectric constant, dissipation factor, dielectric strength, corona, ease of manufacture and handling, variation in properties with temperature and frequency, cost, and susceptibility to pinholes and thickness variation.

4.1.1.4 Furthermore, the contractor shall propose preliminary candidate dielectric systems utilizing the recommended improved materials for pads suitable for use in capacitors satisfying the requirements detailed in section 4.3.

4.1.1.5 For each of the proposed dielectric systems of paragraph 4.1.1.4, the contractor shall estimate the average dielectric constant, average dielectric stress, and energy density (joules/pound) expected in the capacitor pad. In the estimates, the contractor shall take into account the advances made during the Hughes Aircraft program (see paragraph 3.1) in the areas of wrinkle free winding, impregnant purification, and foil edge treatment.

4.1.1.6 Materials classified as polychlorinated biphenyls (PCB's) shall not be considered for use in this program.

4.1.1.7 The end use of the materials shall be considered at all times. For example, if a material has desirable dielectric properties but can not be fabricated in a form suitable for use in capacitors, it shall not be considered for further development.

4.1.1.8 In the modification or development of any material, compatibility with the other materials of the dielectric system (i.e., films, impregnants, papers, and foils) shall be considered.

4.1.2 Task II: Material Test Plan

4.1.2.1 The contractor shall devise a plan for evaluating the materials identified for modification and/or development in the proposal. This test plan shall include a discussion of the quantities of material to be produced, the parameters to be measured, and the instrumentation required.

4.1.2.2 For purposes of comparison, standard tests may be used to evaluate material properties. To the greatest extent possible, however, parameter measurements shall be performed at conditions representative of what the material would experience in its particular capacitor application. For example, dissipation factor and dielectric constant should be measured at the frequencies of interest as determined from the particular capacitor application.

4.1.2.3 The test plan shall include recommended tests to demonstrate compatibility with the other materials of the dielectric system.

4.1.2.4 This test plan shall be submitted for approval by the Air Force project engineer prior to the contractor proceeding with any material fabrication and testing but not later than six (6) months after contract award.

4.1.3 Task III: Material Fabrication, Testing, and Final Selection

4.1.3.1 The contractor shall fabricate samples of the materials identified in section 4.1.1 and suitable for testing in accordance with the approved Material Test Plan as specified in section 4.1.2.

4.1.3.2 The process by which the materials are produced shall be fully documented such that they can be reproduced by others if so desired.

4.1.3.3 Materials shall be fabricated in a form suitable for use in capacitors. For example, the width and thickness of films, papers, and foils shall be representative of the material dimensions to be used in capacitor pads to meet the specifications of section 4.3.

4.1.3.4 The materials shall be tested in accordance with the approved test plan of section 4.1.2.

4.1.3.5 Throughout this phase of the effort, the contractor shall continuously evaluate the various materials and dielectric systems identified in his proposal in order to determine the most promising candidate(s) for each of the four applications specified in section 4.3.

4.1.3.6 The contractor shall give an oral presentation approximately midway through this phase of the program. At this time, the contractor shall report on the status of the program, identify any unforeseen problems which have developed, and propose any changes to the original program plan as required. This presentation shall take place at the AFAPL not later than twelve (12) months after contract award.

4.1.3.7 A second oral presentation shall be required at the conclusion of this task. In addition to the type of information required in the first presentation as specified in paragraph 4.1.3.6, the contractor shall present his recommended dielectric system for each of the four specified capacitor applications to include revised estimates of the characteristics listed in paragraph 4.1.1.5. This presentation shall take place at the AFAPL not later than eighteen (18) months after contract award.

4.1.3.8 Both oral presentations shall require concurrance by the Air Force project engineer prior to presentation.

4.1.3.9 A Material Test Report describing this phase of the program shall be included as part of the Interim Technical Report.

4.2 PHASE II: CAPACITOR PAD DESIGN AND TESTING

4.2.1 Task I: Capacitor Pad Design and Test Plan

4.2.1.1 The contractor shall submit detailed pad designs to meet the capacitor requirements specified in section 4.3.

4.2.1.2 These designs shall include identification of all materials used, any special processing required, quality control procedures, special fabrication techniques employed, drawings, estimates of energy density, and any other information necessary for duplication of these parts.

4.2.1.3 In the designs, the contractor shall consider the improvements in winding, impregnant purification, edge treatment, and other design and fabrication techniques developed under a previous contract as discussed in paragraph 3.1.

4.2.1.4 The contractor shall prepare a test plan for the test and evaluation of capacitor pads fabricated according to the approved designs.

4.2.1.5 The test plan shall be designed such that each pad shall be tested under conditions representative of what it would experience in actual service in capacitors meeting the requirements specified in section 4.3.

4.2.1.6 The pad designs and test plan shall be submitted for approval by the Air Force project engineer through the contracting officer, not later than twenty-one (21) months after contract award.

4.2.2 Task II: Capacitor Pad Fabrication and Testing

4.2.2.1 The contractor shall fabricate capacitor pads in accordance with the approved designs and suitable for testing according to the approved test plan.

4.2.2.2 In the fabrication of these pads, the contractor shall apply any techniques discussed in paragraph 4.2.1.3 and deemed applicable and beneficial to the performance of these componenets.

4.2.2.3 The capacitor pad testing shall include a failure analysis on any pad which does not successfully complete the testing. The contractor shall identify the cause of failure and recommend appropriate action to prevent its recurrence.

4.2.2.4 An oral presentation shall be required approximately nine (9) months into this task but not later than thirty (30) months after contract award. At this time, the contractor shall review the status of the contract and identify any problems associated with meeting projected performance parameters as estimated according to paragraph 4.1.3.7 This presentation shall take place at the AFAPL and shall require concurrence by the Air Force project engineer prior to presentation.

4.2.2.5 A Capacitor Pad Test Report describing this phase of the program shall be included as part of the Final Technical Report.

4.3 CAPACITOR SPECIFICATIONS

4.3.1 High Repetition Rate Pulse Capacitors

4.3.1.1 Capacitors for use in high rep rate pulse forming networks with the following characteristics shall be considered.

PFN Energy Storage	1.5 kilojoules
Number of LC Sections	6
PFN Voltage	15kv
PFN Total Capacitance	13.2µF
PFN Total Inductance	7.6µH
PFN Impedance	0.76Ω
Discharge Current	20ka
Pulse Shape	Rectangular
Pulse Width (90% to 90% amplitude)	20µsec
Rise Time (10% to 90% amplitude)	2µsec
Fall Time (90% to 10% amplitude)	4µsec
Pulse Repetition Rate	300pps
Duty Cycle	1 min on/≤ 2 Hrs off
Life (number of pulses)	10^6
Shelf Life	>1 Yr
Ambient Temperature	160°F max.

4.3.1.2 Additional capacitor characteristics desired for components intended for use in the PFN specified in paragraph 4.3.1.1 are:

Capacitor Energy Density Goal	$\geq$200 joules/lb
	$\geq$ 3j/cu. in.
Energy Stored Per Capacitor	250 joules
Capacitor Voltage Reversal	$\geq$20%
Capacitor Inductance	$\leq$20nH
Capacitance	2.2μF

4.3.2 Low Repetition Rate Pulse Capacitors

4.3.2.1. Capacitors for use in low rep rate pulse forming networks with the following characteristics shall be considered:

PFN Energy Storage	10.56 kilojoules
Number of LC Sections	6
PFN Voltage	40kv
PFN Total Capacitance	13.2μF
PFN Total Inductance	7.6μH
PFN Impedance	0.76Ω
Discharge Current	52.6kA
Pulse Shape	Rectangular
Pulse Width (90% to 90% amplitude)	20μsec
Rise Time (10% to 90% amplitude)	2μsec
Fall Time (90% to 10% amplitude)	4μsec
Pulse Repetition Rate	50 pps
Duty Cycle	1 min on/$\leq$ 2 Hrs Off
Life (number of pulses)	10^6
Shelf Life	$\geq$1 Yr.
Ambient Temperature	160°F max.

4.3.2.2 Additional capacitor characteristics desired for components intended for use in the PFN specified in paragraph 4.3.2.1 are:

Capacitor Energy Density Goal	$\geq$500 joules/pound,
	$\geq$ 10 joules/cu. in.
Energy Stored Per Capacitor	1.76 kilojoules
Capacitor Voltage Reversal	$\geq$20%
Capacitor Inductance	$\leq$20nH
Capacitance	2.2μF

4.3.3 High Voltage Filter Capacitors

Capacitors with the following characteristics for use in high voltage filter applications shall be considered:

Capacitance	0.4μF
Working Voltage	25kv
Ripple	21V_{rms}
Ripple Frequency	20KHz
Current	5.3A_{rms}

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Duty Cycle	Continuous
Dissipation Factor	<0.2%
Energy Density Goal	100 joules/pound

4.3.4 High Frequency AC Capacitors

High frequency AC capacitors satisfying the following requirements for use in series resonant inverters shall be considered:

Capacitance	8µF
Current	480A$_{rms}$
Frequency	10KHz
Voltage	1.5kv peak
Duty Cycle	Continuous
Dissipation Factor	<0.01%
Energy Density Goal	>50 joules/pound

5.0 REPORTS, DATA AND OTHER DELIVERABLES

All data items, including the final report, shall be submitted in accordance with the attached Contract Data Requirements List, DD Form 1423.

6.0 SPECIAL CONSIDERATIONS

In the conduct of the study and selection of techniques, methodology, and materials consideration is to be given to the factors of reliability and maintainability. Reliability/maintainability studies shall be based on sound practical engineering judgment, experience, and available test data. No reliability testing program need be undertaken as part of this evaluation. A prediction of the degree of reliability and maintainability inherent in the techniques (on a basis at least relative to existing devices or subsystems performing a like function) and that which may be achieved through further development, shall be included with an explanation in the final report.

APPENDIX B

MATERIAL TEST PLAN

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│ **HUGHES**   │
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HUGHES AIRCRAFT COMPANY
AEROSPACE GROUPS
CULVER CITY, CA 90230

ADVANCED CAPACITORS

Contract F33615-79-C-2081

Material Test Plan

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1.0 INTRODUCTION

As described in the Statement of Work, the Material Test Plan is clearly intended to perform several different specific functions. It is intended to allow the assessment of improvement in the materials being developed, during the development phase. It is intended to identify and evaluate parameters of particular interest to the capacitor designer, during and at the end of the material development phase. Finally, it is intended to provide specific acceptance test criteria, to be applied as an incoming inspection to the materials as produced in their final forms.

Because of the multiplicity of functions, not all tests described in this document would be performed during a single test sequence. Therefore, schedules are provided relating the individual tests to the specific test sequence. Not all tests are intended to provide a "go-no go" criterion, but rather to provide parametric values of the various material properties of interest to the capacitor designer. For these tests, the word "Report" appears in place of a specific set of limits for acceptance and rejection.

This test plan is not organized along the lines of a military specification, although with a little work a document of that sort could be produced. This document is simply a test plan for the use of people developing specific sets of materials, and is not intended to be and should not be used as a specification.

2.0 TYPES OF TEST SEQUENCES

As pointed out in the Introduction, three specific test sequences are envisioned. This Section describes these test sequences. Except where otherwise noted, prototype quantities of insulating materials are required for each test; these materials should be characteristic of the production run, if any, from which they were taken.

2.1 DEVELOPMENTAL TESTING

Developmental testing is performed during the work aimed at improving the characteristics of insulating materials. Examples would be tests such as non-destructive fault counts of capacitor film during the effort to clean up the film and remove any particulate contamination, or chemical analysis of insulating oils during experiments to determine the most effective oxidation inhibitors. Therefore, some particularly difficult or delicate tests may be included, tests that would not normally be performed during incoming inspection, for example. Complete parametric characterization would not normally be performed at this stage unless new materials, new chemical compounds, or new mixtures of compounds were being used.

Developmental tests are those tests performed at the discretion of the development engineer to show improvement; only the properties which are expected to improve would normally be tested.

2.2 PARAMETRIC CHARACTERIZATION

Tests to completely characterize the material may be performed during the development phase, but most certainly will be performed at the end of the development phase.

The purpose of this battery of tests is to give information
to the capacitor designer. Therefore, measurements of im-
portant properties at the anticipated operating points in
actual devices will be made. Tests not directly connected
with capacitor design (e.g., some of the mechanical properties)
would not be made. If the particular insulation material is
one upon which measurements have previously been made and
reported, measurements would only be made at the operating
points, unless the accuracy of the previous measurement was
in doubt. An example would be the polysulfone, for which
it has been proposed to further clean up the manufacturing
process so that a higher dielectric strength may be achieved.
In this instance, dielectric strength would be measured, but
the previously reported data on parameters such as dielectric
constant would be used where available.

2.3 ACCEPTANCE TESTING

Acceptance tests are those performed to make reasonably
certain that the insulation material is as ordered, to detect
gross defects, and to make sure the material has been pro-
perly packaged and not damaged in shipment. For these rea-
sons, neither complete parametric characterizations nor
difficult developmental tests will be performed. Many
acceptance tests are performed on full rolls of capacitor
film or entire drums of insulating oil, as noted.

3.0 TEST PROCEDURES FOR FILMS

These tests will be performed on polymeric film intended for use in capacitors. Normally this film will have a thickness of 12 micrometers (0.0005 inch) or less, but in any case it will be thinner than 125 micrometers (0.005 inch). Film is normally supplied in standard bobbins or rolls; details of the packaging (put-up) are to be found in the subsection on packaging.

3.1 ELECTRICAL TESTS

These tests allow electrical characterization of the film. In some cases they may be performed at a series of different frequencies and temperatures; this depends upon the specific application envisioned, and is noted in the tables in Section 6.

3.1.1 <u>Dielectric Constant</u>. Testing is performed per ASTM Specification D 150-78, using a Balsbaugh Research Cell with guarded electrodes, and the two fluid technique employing room air and silicone oil (such as DC-200).

3.1.2 <u>Dissipation Factor</u>. This test is performed per ASTM Specification D 150-78 using the same technique as for the dielectric constant determination, paragraph 3.1.1.

3.1.3 <u>Surface and Volume Resistivity</u>. The test is performed per ASTM Specification D 257-78, for 60 s using 100 VDC.

3.1.4 <u>Dielectric Strength</u>. These tests are performed per ASTM Specification D 149-75 and D 2305-72. The requirement for the 2 inch diameter electrodes is waived, but all tests performed on a given material must be made with the same diameter electrodes and the same electrode pressure. The

size and type of electrodes and the electrode pressure must
be reported. Use a rate of rise of 500 V/s, and make ten
separate measurements and average the results.

3.1.5 Gross Flaw. This test can be performed by one of two
methods: a standard mercury electrode clearing test, or the
"stain test". In the stain test, denatured alcohol is care-
fully wiped over an area of film beneath which is a sheet of
"Hectograph" or "Mimeograph" paper (the deep blue or black
reproduction master, with the colored or ink side placed
uppermost). At least 0.5 square feet shall be tested for
each sample. An average of at least 5 measurements is re-
quired. Faults are counted where the alcohol causes a "stain"
on the film by the ink from the Hectograph paper bleeding
through.

3.1.6 Fine Flaw. This test is to be performed using the
destructive testing apparatus developed by Schweitzer on this
program. At least 0.5 square feet of film shall be tested
for each sample. An average of 5 samples is required. The
test voltage is to be specified. This test conforms in
most respects to ASTM Specification D 202, Method A.

3.2 TESTS FOR CONTAMINANTS

These tests are primarily designed to find contaminants
added either during the production of the insulation (such as
solvent) or in the handling after production (such as oil
or particulate matter). The data obtained by these tests is
important in interaction with the insulation supplier and in
the design of the fabrication and drying processes.

3.2.1 Surface Contamination. This test is to be performed
according to ASTM Specification D 202-77, paragraphs 45-52,
except that a 5 g specimen and 200 ml of boiling distilled water

are to be used. Omit the stirring. After the filtration
step wash the specimen with 50 ml hot distilled water and
add it to the filtrate. Adjust the total volume to 250 ml
with hot distilled water. A Balsbaugh liquid cell may be
used in place of the platinum electrode cell.

3.2.2 Residual Solvent. An accurately weighed sample of
about 1 g of film is placed in an oven at 160° C for 30
minutes, and its loss in weight is measured.

3.2.3 Moisture Absorption. This test is to be performed
according to ASTM Specification D 570.

3.3 PHYSICAL AND MECHANICAL TESTS

These tests measure two types of parameters: proper-
ties of the material that a designer might need to know,
such as density and thickness; and proerties that are the
result of material preparation, such as tensile strength,
which may be needed for the design of the assembly process.

3.3.1 Film Thickness. Two different methods are used, and
either is allowable, but the test report must state which
method was used.

3.3.1.1 Roll Weight Method. Calculate the average thick-
ness from the average density, measured according to para-
graph 3.3.2, and the width, length, and net weight of the
roll.

3.3.1.2 Ten Sheet Stack Method. Use either Method A or
Method C of ASTM Specification D 374. Make measurements on
a ten-sheet stack of film from a single roll. The micro-
meter foot may not be any closer than 19 mm to a folded
edge or 6 mm from a cut edge.

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3.3.2 <u>Film Density</u>. The density of the capacitor film shall
be determined by ASTM Specification D 1505. Carbon tetra-
chloride and n-heptane shall be used for the density gradient
column. WARNING. Carbon tetrachloride is highly toxic and
n-heptane is flammable. Technicians shall take appropriate
precautions.

3.3.3 <u>Tensile Strength at Break</u>. This test is to be per-
formed in accordance with ASTM Specification D-882, Method
A. The equipment shall be a constant strain rate tester,
Thwing-Albert tensile tester or equivalent. The specimen
size shall be 15 mm x 200 mm, and the specimen must be most
carefully cut, to avoid edge nicks or cuts. The initial
jaw separation shall be 100 mm, and the elongation rate
shall be 1.0 mm/s (approximately 2 inches per minute). The
value for tensile strength is calculated from the cross-
sectional area of the specimen and this, in turn, is related
to the specimen thickness. Thickness should be measured
according to paragraph 3.3.1.1. A minimum of five measure-
ments must be averaged to obtain a reportable value.

3.3.4 <u>Elongation at Break</u>. This data is obtained from exam-
ination of the charts produced during the tensile strength
test, paragraph 3.3.3.

3.3.5 <u>Shrinkage</u>. Make a pair of marks 100 mm apart exactly,
on a specimen that is approximately 150 mm long. Suspend
the specimen in a oven at 135°C for two hours. Allow the
sample to cool to room temperature and measure the distance
between the two marks. Different temperatures may be used
as required, but the temperature must always be reported.

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3.4 ROLL FORMATION AND CHARACTERISTICS

Capacitor material is supplied in various roll sizes, and the rolls are packaged in various ways. The purpose of these specifications and tests is to ensure that the packaging and put-up does not damage the material or make it difficult or impossible to wind into capacitors.

3.4.1 Cores. The cores upon which the capacitor film is wound must not distort or collapse from the tension of the film wound thereon, nor must they flake or degrade the capacitor film. The film must be evenly spaced on the core, with neither side of the core recessed or extended more than 0.8 mm (0.03125 inch or 1/32 inch). The inside diameter of the cores may be any of the three following values:

> 1-15/16 inches +1/16-0.0 inch
> 3.0 + 0.0625 inches
> 5-15716 inches + 1/16 inch

In general, for small rolls or rolls narrower than 6 inches the smallest bobbin is preferred. The largest bobbin is normally supplied on large widths and converter rolls (rolls that the capacitor manufacturer is intending to slit or otherwise reprocess.).

3.4.2 Roll Workmanship. It is necessary for the rolls to be extremely uniform for quality capacitors to be manufactured from them. The following requirements apply:

3.4.2.1 Telescoping. The rolls are not dished by more than 1.6 mm (0.0625 inch) from the plane of the edge of the roll.
3.4.2.2 Shiners. There shall be no more than three shiners per roll, and none extending more than 1.6 mm (0.0625 inch). (A shiner is a single or multiple layer of film extending beyond the edge of the slit roll, which if folded over displays a shiny appearance.)

bands, wrinkles and creases, extensive scratches, or obvious changes in color.

3.4.3 Width and Diameter. The tolerances on the width and diameter of any roll shall be as follows:

Width	Tolerance
<3 inch	$\pm$ 0.016 inch
3 to 9 inch	$\pm$ 0.031 inch
>9 inch	$\pm$ 0.0625 inch

Diameter	Tolerance
All	$\pm$0.5 inch

The diameter of the finished slit rolls shall be as agreed upon between the capacitor manufacturer and the film supplier, except that no roll shall have a diameter larger than eight inches.

3.4.4 Splices. All splices shall be carefully made using the overlapping type of splice and pressure sensitive polyester tape. The material shall be chosen to minimize strikethrough to adjacent layers and oozing at the edges of the rolls. The maximum number of splices shall be three per roll except for film thinner than 9 gauge (0.09 mil), where the maximum number of splices shall be 5. No splice may be closer than 300 feet to either end of the roll or to another splice. Films produced on an experimental basis may have the number of splices agreed to between the film maker and the capacitor manufacturer.

3.4.5 Marking. Each roll shall be marked on the core or on a label placed on the inside diameter of the core. The marking shall include: thickness, mill roll number, manufacturer's designation, and material.

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3.5 PACKAGING

These specifications are designed to protect the material during its transit to and handling at the capacitor manufacturer's plant. It is important to keep the film clean and undamaged.

3.5.1 Wrapping. All rolls shall be wrapped in a non-fibrous material. Slit bobbins of film shall subsequently be heat-sealed in polyethylene bags.

3.5.2 Outer Package. Rolls are boxed in corrugated paper containers. Converter rolls are core supported.

3.5.3 General Requirement. The capacitor material shall be packaged in standard commercial containers designed to protect the roll from damage, and constructed to ensure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery, unless otherwise specified.

4.0 TEST PROCEDURES FOR OILS

These tests and specifications govern the use of insulating oils as capacitor impregnants. It is intended that this section be limited to liquids useful as insulants, and therefore no consideration is given to the insulating properties of various gasses. Further, since the use of the class of halogenated aromatic hydrocarbons known as polychlorinated biphenyls has been prohibited in the United States, these tests and procedures apply to such materials only incidentally.

4.1 PHYSICAL PROPERTIES

Tests described below produce data necessary in the design of processing procedures and also testify to the purity of the insulant.

4.1.1 Viscosity. The viscosity of the oil shall be measured in accordance with ASTM Specification D-88 to obtain the Saybolt viscosity, and in accordance with ASTM Specification D-445 to obtain the kinematic viscosity.

4.1.2 Flash and Fire Points. The flash and fire points of the oil shall be determined according to the ASTM Specification D-92, except that a temperature of 300°C shall be the upper test limit.

4.1.3 Pour Point. The pour point of the insulation shall be determined in accordance with ASTM Specification D-97.

4.1.4 Specific Gravity. The specific gravity shall be determined according to the methods of ASTM Specification D-1898.

4.1.5 Weight per Gallon. The weight per gallon may be obtained from the value measured for the specific gravity or it may be measured directly using a laboratory scale.

4.1.6 Interfacial Tension. The interfacial tension of the fluid shall be determined in accordance with ASTM Specification D-971.

4.1.7 Color. The color of the insulating oil as referred to the ASTM color scale shall be determined according to ASTM Specification D-1500.

4.1.8 Visual Examination. The oil shall be visually examined and its quality shall be estimated in accordance with the rules contained in ASTM Specification D-1524.

4.2 ELECTRICAL PROPERTIES

These tests produce information of interest to the designer for the calculation of field balances, and at the same time ensure that the quality of the material is sufficient for use in high quality electrical components.

4.2.1 Dielectric Constant. The dielectric constant shall be measured using the procedures set forth in ASTM Specification D-924. The choice of test cells shall be limited to those shown in Figures A2 and A3, and the same cell shall be used for all determinations.

4.2.2 Dissipation Factor. The dissipation factor of the oil shall be measured using the same procedure and equipment as described in paragraph 4.2.1, except that the preferred cell shall be that shown in Figure A2, only.

4.2.3 Dielectric Breakdown Voltage. Two different types of tests are in use, and either may be used provided that the

type of test is reported along with the test value. A
particular application may require one or the other, as
they are not identical, nor do they give the same results.
In this case, the type of test will be called out.

4.2.3.1 Power Frequency Test, Disk Electrodes. The break-
down voltage shall be determined using disk electrodes in
accordance with ASTM Specification D-877.
4.2.3.2 Power Frequency Test, VDE Electrodes. This test
is much more sensitive to contaminants than is the D-877
test. The dielectric breakdown strength shall be deter-
mined according to ASTM Specification D-1816.

4.2.4 Volume Resistivity. The volume resistivity of the oil
shall be determined using a standard Balsbaugh test cell
and a very stable DC power supply. The voltage used shall
be 100 VDC. The volume resistivity of the oil shall be
determined using the measured current and the cell constant
provided with the particular cell.

4.3 CHEMICAL PROPERTIES

These tests assess the possible chemical contaminants
that may adversely affect the life of the insulation system,
and also, in some cases, measure compounds that may have
been deliberately added, e.g., to improve the oil life under
oxidizing conditions.

4.3.1 Neutralization Number. This test measures the acid
content of the oil. A very low acid content is necessary to
avoid attack on other parts of the capacitor system. The
neutralization number shall be measured per ASTM Specifi-
cation D 974, and shall be less than 0.05 mg KOH/g unless
otherwise specified.

4.3.2 Water Content. The presence of water in insulating oil will cause degradation of the insulating system in service, particularly for systems containing mineral oil, silicate esters, or kraft paper. However, the amount of water in the oil in a as-received condition is much less important than the actual water in the oil at the time it is placed in the capacitors. Therefore, this test must be performed on specimens collected during the capacitor impregnation process to ensure that a low water content was achieved by the oil processing steps. The water determination may be made by the Karl Fischer titration method either manually or using one of the commercially available instruments such as the Photovolt Aquatest. For reference and procedures refer to ASTM Specification D 1315 or D 1533. It is important to note that there is a sensitivity limit to this process, and therefore some care must be used in interpreting results lower than about 20 ppm.

4.3.3 Inorganic Chlorides and Sulfates. This test measures a certain set of species of chemical contaminants; it should not be used for materials such as polychlorinated biphenyls, which are already known to contain some free chlorides or sulfates. Perform this test per ASTM Specification D 878.

4.3.4 Oxidation Stability. This test is used to measure the resistance of the fluid to oxidation, and incidentally to verify the presence of an oxidation inhibitor where one has been added. There are two different types of tests: the sludge test and the rotating bomb test. Tests should be performed per ASTM Specification D 2112 if a quick check is desired on a fluid known to contain oxidation inhibitors. Otherwise, tests should be performed using the acid-sludge method given is ASTM Specification D 2440.

4.3.5 Oxidation Inhibitor Content. Various materials are routinely added to insulating oils to prolong the life of the insulation system and retard degradation. The material added must of course prevent the specific mechanism associated with the particular oil. Thus, tin tetraphenyl was added to polychlorinated biphenyl to scavenge the free chlorine and prolong life. In mineral oils, two particular chemical compounds are commonly used: 2,6 Ditertiary Butyl Para-Cresol, and 2,6 Ditertiary Butyl Phenal. This test is designed to detect the presence of these two compounds. Perform the test per ASTM Specification D 2668.

4.3.6 Gassing. Many of the mechanisms postulated to account for failure in capacitors rely upon the gassing of the impregnant to form the first breakdown or corona site. Therefore, some emphasis has recently been placed upon the selection of fluids with reduced gassing tendency. This test measures that property. Perform the test per ASTM Specification D 2300.

4.4 PARTICULATE CONTAMINATION

It has become increasingly clear that all liquid insulating materials must be as clean as possible, and must be maintained as clean as possible. Therefore, it is necessary to test for particles in the oils as part of routine examinations. Two methods may be used: direct examination of the filters, or by machine. For the direct method, a capillary-type filter membrane should be used, the final stage capable of trapping particles as fine as 1.0 µm. This test may be performed as an incoming inspection, and should be performed in conjuction with the water and resistivity tests as a quality control on each capacitor batch.

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5.0 TEST PROCEDURES FOR PAPERS

Kraft capacitor tissue is a very old and well known
material, although in the last twenty years the manu-
facturers have greatly improved many of its properties.
There are numerous specifications for this material, on
account of its wide and persistent use. This section
relies primarily upon the information given in the ASTM
Standard Specification for Kraft Dielectric Tissue,
Capacitor Grade, D-1930-74, with additions from the ex-
perience of the manufacturer and the supplier. Many
test methods are given in ASTM/ANSI Specification D-202.

5.1 ELECTRICAL PROPERTIES

These tests allow electrical characterization of the
paper. They are substantially more difficult and involved
than the tests described previously for films, for the reason
that some of these tests must be performed in conjunction
with the contemplated impregnant, and in all cases great
care must be taken to maintain the paper in a bone-dry
condition. In some cases the tests may be performed at a
series of different frequencies and temperatures; this
depends upon the specific application envisioned.

5.1.1 Dielectric Constant. The dielectric constant of the
kraft is influenced by its apparent density, that is, the
amount of air that is also present between two measuring
electrodes. This test is therefore difficult and often
not particularly conclusive, since the dielectric constant
may be calculated from the apparent density and the dielectric
constant of cellulose. Tests in the dry or unimpregnated
state should be performed according to ASTM Specification
D 150, using a Balsbaugh research cell with guarded elec-
trodes. Two fluid or impregnated measurements can be made
using the same cell and Specification D 2413.

5.1.2 Dissipation Factor. Dissipation factor is subject to the same comments made above in section 5.1.1. Measurement may be made dry according to ASTM Specification D-150, and with fluid according to ASTM Specification D-2413. Specification D-202 should be consulted also for material preparation.

5.1.3 Dielectric Strength. This measurement should be made on dry paper in accordance with ASTM Specification D 202, parts 134-139. On impregnated paper, it should be made in accordance with ASTM Specification D-149 and D-2413.

5.1.4 Conducting Paths. Measurement to determine the number of conducting paths in the material per unit area should be made in accordance with ASTM Specification D-202, parts 121 to 133.

5.1.5 Fine Flaws. Testing for very fine pinholes and other such flaws may be performed using the standard 'stain test', as described in section 3.1.6 of this document or in ASTM Specification D-202, parts 166-192.

5.2 PHYSICAL TESTS

These tests measure two type of parameters: properties of the material that a designer might need to know, such as density and thickness; and properties that are the result of material preparation and might be used as quality control tests, such as tensile strength.

5.2.1 Paper Thickness. Two different types of methods are used, and either is allowable, but the test report must state which type of method has been used.
5.2.1.1 Roll Weight Method. Calculate the average thickness from the average density, measured according to paragraph 5.2.2, and the width, length, and net weight of the roll.

5.2.1.2 Ten Sheet Stack Method. Use either Method A or Method C of ASTM Specification D 374. Make measurements on a ten sheet stack of paper from a single roll. The micrometer may not be any closer than 6 mm from a cut edge.

5.2.2 Apparent Density. The apparent density of a paper is a measure of the surface roughness and porosity; the apparent density is always less than the nominal density of cellulose, and a qualitative estimate of the roughness may be obtained by comparing the apparent density to the density of cellulose. Kraft paper is commonly made in a series of nominal apparent densities ranging from 0.90 to 1.20. Determine the apparent density by Method A of ASTM Specification D 202, parts 28-29.

5.2.3 Holes and Felt Hair Inclusions. Occasionally, because of the manufacturing process and the nature of the raw materials, kraft paper may have visible pinholes and undesirable solid inclusions. Test for these per ASTM Specification D 202, parts 86-91. Ignore the limitation to type 1C paper.

5.2.4 Tensile Strength and Yield Properties. Test the paper for tensile strength and yield properties according to ASTM Specification D202, parts 102-112, and Specification D 76.

5.2.5 Air Resistance. This test measures relative porosity of the paper. Determine the air resistance in accordance with Method C of ASTM Specification D 726.

5.3 CHEMICAL TESTS

There are a large number of tests that one might perform on kraft paper and on the pulps from which the paper

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is produced. Many of these test were originally designed for use in the laboratories of the paper-making companies, and the results they provide are, from the point of view of the user, interesting but of no practical use. Therefore, only those tests which will provide data to help quality control or to enlighten the capacitor designer are given in this section.

5.3.1 Aqueous Extract Conductivity. The conductivity of the water extract of kraft paper results from electro- lytic impurities in the paper which may be present as ionizable acids, bases, salts, or a combination of these. They are usually incompletely removed chemical residues of the paper manufacturing process. Their presence in excessive quantities has been shown to greatly shorten the useful life of capacitors. Test for aqueous extract conductivity according to ASTM Specification D 202, parts 45-52. A Balsbaugh liquid cell may be used in place of the cell specified.

5.3.2 Soluable Chlorides. This test detects soluable chlorides that are present in the paper. The presence of such impurities is undesirable, and, if not countered by an appropriate scavenger in the impregnation fluid, will greatly shorten the life of capacitors made from such paper. Test for soluable chlorides according to ASTM Specifi- cation D 202, parts 148-165.

5.3.3 Acidity-Alkalinity-pH. This test detects residual chemical species left over from the manufacturing process. Test for pH according to ASTM Specification D 202, parts 36 to 44, and Specification E 70.

5.3.4 Moisture Content. It is important to know the moisture content of the paper as it is received from the manufac- turer, so that proper conditions may be provided for its

storage. It is well known that if a roll of kraft paper
is placed in an environment where the air has either more
or less moisture than the air with which the roll is in
equilibrium, the ends of the roll will tend to swell or
shrink, respectively, and this causes irreversable wrinkles
and folds in the paper which are later found to be sites of
corona damage. Kraft paper is normally shipped with a
moisture content that is in equilibrium with 50% relative
humidity, and therefore the paper must be stored and wound
in that humidity to avoid deleterious effects. Test for
moisture content according to ASTM Specification D 202,
parts 30-32, and Specification D 644.

5.4 ROLL CHARACTERISTICS AND PACKAGING

Capacitor paper is supplied in a wide variety of roll
sizes and widths, and the rolls are packaged in different
ways. The purpose of these specifications and tests is to
make sure that the put-up and packaging does not damage the
paper, that the put-up is compatible with common winding
methods, that the material is supplied to the correct width,
and that the packaging adequately protects the material
during shipment from the paper mill to the capacitor plant.

5.4.1 Cores. The cores upon which the capacitor paper is
wound must not distort or collapse from the tension of the
paper wound thereon, nor must they degrade the paper by
contributing impurities or by any other means. The paper
must be evenly spaced on the core, with neither side of
the core recessed or extended more than 0.8 mm (1/32 inch).
The inside diameter of the core may be either 3.0±0.0625 inches,
or 1.125+0.0625-0.0 inches. In general, the larger roll
is preferred. The normal outer diameter supplied is 6 inches
for the small roll and 8 inches for the large roll.

5.4.2 Roll Workmanship. The workmanship of the roll shall
conform to the requirements of ASTM Specification D-1930,
paragraph 8, or the manufacturer's standard specifications,
whichever is more stringent.

5.4.3 Width. Tolerances on all widths shall be $\pm$ 0.015
inch unless otherwise agreed between the manufacturer and
the user.

5.4.4 Marking. The marking requirements of ASTM Specification
D 1930 shall apply.

5.4.5 Packaging. The packaging requirements of ASTM Speci-
fication D 1930 shall apply.

6.0 REQUIREMENTS

This Section contains tables that give required test points and acceptance values for each of the tests described in the three preceding Sections, for each of the three different test sequences described in Section 2. Table 6-1 gives the various combinations of temperature and frequency that an analysis of the statement of work has shown to be necessary. Each combination or group of combinations is assigned a letter abbreviation for reference on the succeeding tables. Table 6-2 gives the requirements for the Developmental phase. Table 6-3 gives the requirements for Parametric Characterization. Table 6-4 gives the requirements for Acceptance Testing.

6.1 Measurement Points. Table 6-1 gives the combinations of measurement points that are referred to in succeeding tables.

Table 6-1. Measurement Points

Index	Measurement Points
A	25°C, 1 kHz
B	25°C, laboratory environment
C	(25°C, 85°C)X(1kHz, 2kHz, 5kHz, 10kHz, 20kHz, 50kHz, 100kHz).
D	(25°C, 85°C, 125°C)X(1kHz, 2kHz, 5kHz, 10kHz, 20kHz, 50kHz, 100kHz)
E	(25°C, 85°C)X(10kHz, 20kHz, 50kHz, 100kHz)
F	(25°C, 85°C, 125°C)X(10kHz, 20kHz, 50kHz, 100kHz)
G	1kHz, (-55°C, -25°C, 0°C, 25°C, 85°C, 105°C).
H	DC, (25°C, 85°C, 105°C)
I	DC, (125°C, 150°C)

6.2 Requirements for Development

Table 6-2. Developmental Requirements

Paragraph	Item	Requirement	Index
3.1.1	Diel. Constant	Report	A
3.1.2	DF	Report	A
3.1.3	Surf.,Vol.Res.	Report	B
3.1.4	Diel.Strength	Report	B
3.1.5*	Gross Flaw	1 per square foot	B
3.1.6	Fine Flaw	Report	B
3.2.1	Surf. Contam.	Report	B
3.2.2	Resid. Solvent	Report	B
3.2.3	Moist. Absorb.	Report	B
3.3.1	Thickness	Report	B
3.3.2	Density	Report	B
3.3.3*	Tensile	Report	B
3.3.4*	Elongation	Report	B
3.3.5*	Shrink.	Report	B
4.1.1*	Viscosity	Report	B
4.1.2*	Flash+Fire	As good as mineral oil	B
4.1.3	Pour Pt.	Report	B
4.1.4	Spec. Grav.	Report	B
4.1.5	Weight	Report	B
4.1.6*	IFT	Report	B
4.1.7*	Color	Report	B
4.1.8	Visual	Clear and bright	B
4.2.1	Diel. Const.	Report	A
4.2.2	DF	Report	A
4.2.3	BreakdownV	Report	A
4.2.4	Vol. Res.	Report	A

*denotes required measurement.
"Index" refers to Table 6-1.

Table 6-2 Continued.

Paragraph	Item	Requirement	Index
4.3.1*	Neut. Number	less than 0.1mgKOH/g	B
4.3.2*	Water Content	less than 50 ppm	B
4.3.3	Inorg. Chloride	Report	B
4.3.4	Oxid. Stability	Report	
4.3.5	Inhibitor Cont.	Report	
4.3.6	Gassing	Report	B
4.4	Particles	Report	B
5.1.1	Diel. Constant	Report	B
5.1.2	DF	Report	B
5.1.3	Diel. Strength	Report	B
5.1.4*	Conducting Path	7/sq. ft., for 0.3 mil	B
5.1.5	Fine Flaw	Report	B
5.2.1*	Thickness	Nominal $\pm$0.00002 inch	B
5.2.2	App. Density	Nominal $\pm$0.05	B
5.2.3	Holes etc.	per ASTM D 1930	B
5.2.4	Tensile	Report	B
5.2.5*	Air Res.	per ASTM D 1930	B
5.3.1*	Aq. Extract	3.3 micromhos/cm at mfg.	B
5.3.2*	Chlorides	8 ppm	B
5.3.3	pH	6.2 to 7.4	B
5.3.4	Moisture	4.0% to 7.5%	B

Notes: * indicates required test; all others are as needed.

6.3 Requirements for Parametric Characterization.

Table 6-3. Measurement Requirements

Paragraph	Item	Requirement	Index
3.1.1*	Diel. Constant	Report	C,D,E,F, or G
3.1.2*	DF	Report	C,D,E,F, or G
3.1.3*	Surf.,Vol.Res.	Report	H or I
3.1.4*	Diel.Strength	Report	G
3.1.5*	Gross Flaw	1 per square foot	B
3.1.6	Fine Flaw	Report	B
3.2.1	Surf. Contam.	Report	B
3.2.2*	Resid. Solvent	Report, depends on mat'l	B
3.2.3	Moist. Absorb.	Report	B
3.3.1*	Thickness	Nominal $\pm 10\%$	B
3.3.2*	Density	Nominal $\pm 0.1 g/cm^3$	B
3.3.3*	Tensile	Report	B
3.3.4*	Elongation	Report	B
3.3.5*	Shrink.	Report	B
4.1.1*	Viscosity	Report	H
4.1.2*	Flash+Fire	As good as mineral oil	B
4.1.3*	Pour Pt.	Report	
4.1.4	Spec. Grav.	Report	B
4.1.5	Weight	Report	B
4.1.6*	IFT	Report	B
4.1.7*	Color	Report	B
4.1.8*	Visual	Clear and bright	B
4.2.1*	Diel. Const.	Report	C,D,E,F, or G
4.2.2*	DF	Report	C,D,E,F, or G
4.2.3*	BreakdownV	Report	H
4.2.4*	Vol. Res.	Report	H

*denotes required measurement.
"Index" refers to Table 6-1.

Table 6-3 Continued.

Paragraph	Item	Requirement	Index
4.3.1*	Neut. Number	less than 0.1mg KOH/g	B
4.3.2*	Water Content	less than 35 ppm	B
4.3.3	Inorg. Chloride	Report	B
4.3.4*	Oxid. Stability	Report	
4.3.5*	Inhibitor Cont.	If present, nominal +10%	
4.3.6	Gassing	Report	B
4.4*	Particles	Report	B
5.1.1*	Diel. Constant	Report	C,D,E, or F
5.1.2*	DF	Report	C,D,E, or F
5.1.3*	Diel. Strength	Report	H or I
5.1.4*	Conducting Path	3.5/sq. ft. for 0.3 mil	B
5.1.5*	Fine Flaw	Report	B
5.2.1*	Thickness	Nominal +0.00002 inch	B
5.2.2*	App. Density	Nominal +0.05	B
5.2.3	Holes etc.	Report, per ASTM D 1930	B
5.2.4	Tensile	Report	B
5.2.5*	Air Res.	Not to exceed ASTM D1930	B
5.3.1*	Aq. Extract	3.3 micromhos/cm at mfg.	B
5.3.2*	Chlorides	8 ppm	B
5.3.3	pH	6.2 to 7.4	B
5.3.4	Moisture	4.0% to 7.5%	B

6.4 Requirements for Acceptance Testing

Table 6-4. Acceptance Requirements

Paragraph	Item	Requirement	Index
3.1.1*	Diel. Constant	Nominal ±0.1 relative	A
3.1.2*	DF	Nominal max not exceeded	A
3.1.3	Surf.,Vol.Res.	Report	B
3.1.4*	Diel.Strength	Nominal min. or more	B
3.1.5*	Gross Flaw	1 per square foot	B
3.1.6*	Fine Flaw	Report	B
3.2.1	Surf. Contam.	Report	B
3.2.2*	Resid. Solvent	Report	B
3.2.3	Moist. Absorb.	Report	B
3.3.1*	Thickness	Nominal ±5% or specification	B
3.3.2	Density	Report	B
3.3.3*	Tensile	Exceeds minimum spec'd.	B
3.3.4*	Elongation	Nominal ±15%	B
3.3.5*	Shrink.	Less than max spec'd	B
4.1.1*	Viscosity	Nominal ±10%	B
4.1.2*	Flash+Fire	As good as mineral oil	B
4.1.3*	Pour Pt.	Lower than spec'd value	B
4.1.4	Spec. Grav.	Report	B
4.1.5	Weight	Report	B
4.1.6*	IFT	Report	B
4.1.7*	Color	Report	B
4.1.8*	Visual	Clear and bright	B
4.2.1*	Diel. Const.	Nominal ±10%	A
4.2.2	DF	Not to exceed spec'd	A
4.2.3	BreakdownV	Exceeds min spec.	A
4.2.4	Vol. Res.	10^{10} or better	A

*denotes required measurement.
"Index" refers to Table 6-1.

Table 6-4 Continued.

Paragraph	Item	Requirement	Index
4.3.1*	Neut. Number	less than 0.1mgKOH/g	B
4.3.2*	Water Content	less than 75 ppm	B
4.3.3	Inorg. Chloride	Report	B
4.3.4	Oxid. Stability	Report	
4.3.5*	Inhibitor Cont.	Exceeds min. spec'd.	
4.3.6	Gassing	Report	B
4.4	Particles	No requirement	B
5.1.1	Diel. Constant	Report	B
5.1.2	DF	Report	B
5.1.3	Diel. Strength	Report	B
5.1.4*	Conducting Path	3.5/sq.ft. for 0.3 mil	B
5.1.5	Fine Flaw	Report	B
5.2.1*	Thickness	Nominal ±0.00002 inch	B
5.2.2	App. Density	Nominal ±0.05	B
5.2.3*	Holes etc.	Per ASTM D 1930	B
5.2.4	Tensile	Report	B
5.2.5*	Air Res.	Per ASTM D 1930	B
5.3.1*	Aq. Extract	3.3 micromhos/cm at mfg.	B
5.3.2*	Chlorides	8 ppm	B
5.3.3	pH	6.2 to 7.4	B
5.3.4*	Moisture	4.0% to 7.5%	B

Note: In addition the materials must meet the roll packaging
requirements of sections 3.4, 3.5, and 5.4.

7.0 INDEX OF ASTM SPECIFICATIONS

Specification Number	Title
D 88	Test for Saybolt Viscosity
D 92	Test for Flash and Fire Points by Cleveland Open Cup
D 97	Test for Pour Point of Petroleum Oils
D 149	Tests for Dielectric Breakdown Voltage and Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies.
D 150	Tests for AC Loss Characteristics and Permittivity of Solid Electrical Insulating Materials.
D 202	Sampling and Testing Untreated Paper Used for Electrical Insulation
D 257	Tests for DC Resistance or Conductance of Insulating Materials
D 374	Tests for Thickness of Solid Insulation
D 445	Test for Kinematic Viscosity of Transparent and Opaque Liquids
D 570	Test for Water Absorption of Plastics
D 644	Test for Moisture Content of Paper and Paperboard by Oven Drying
D 726	Test for Resistance of Paper to Passage of Air
D 877	Test for Dielectric Breakdown Voltage of Insulating Liquids Using Disk Electrodes
D 878	Test for Inorganic Chlorides and Sulfates in Insulating Oils
D 882	Tests for Tensile Properties of Thin Plastic Sheeting
D 924	Test for Power Factor and Dielectric Constant of Electrical Insulating Liquids
D 971	Test for Interfacial Tension of Oil Against Water by the Ring Method

Specification Number	Title
D 974	Test for Neutralization Number by Color Indicator Titration
D 1298	Test for Density, Specific Gravity, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
D 1315	Test for Water in Insulating Oils by Extraction
D 1500	Test for ASTM Color of Petroleum Products
D 1505	Test for the Density of Plastics by the Density Gradient Technique
D 1524	Visual Examination of Used Electrical Insulating Oils of Petroleum Origin in the Field
D 1533	Test for Water in Insulating Liquids, Karl Fischer Method
D 1816	Test for Dielectric Breakdown Voltage of Insulating Oils of Petroleum Origin Using VDE Electrodes
D 1930	Specification for Kraft Dielectric Tissue, Capacitor Grade
D 2112	Test for Oxidation Stability of Inhibited Insulating Oil by Rotating Bomb
D 2300	Test for Gassing of Insulating Oils Under Electrical Stress and Ionization
D 2305	Testing Polymeric Films Used for Electrical Insulation
D 2413	Preparation and Electrical Testing of Insulating Paper and Board Impregnated With a Liquid Dielectric
D 2440	Test for Oxidation Stability of Mineral Insulating Oil
D 2668	Test for 2,6-Ditertiary-Butyl Para-Cresol and 2,6-Ditertiary Butyl Phenal in Electrical Insulating Oil by Infrared Absorption
E 70	Test for pH of Aqueous Solution with the Glass Electrode

such flaws may be performed using the standard 'stain test', as described in section 3.1.6 of this document or in ASTM Specification D-202, parts 166-192.

5.2 PHYSICAL TESTS

These tests measure two type of parameters: properties of the material that a designer might need to know, such as density and thickness; and properties that are the result of material preparation and might be used as quality control tests, such as tensile strength.

5.2.1 Paper Thickness. Two different types of methods are used, and either is allowable, but the test report must state which type of method has been used.

5.2.1.1 Roll Weight Method. Calculate the average thickness from the average density, measured according to paragraph 5.2.2, and the width, length, and net weight of the roll.

END